

The Journal *of the* Society of Dyers and Colourists

Volume 77



Number 11

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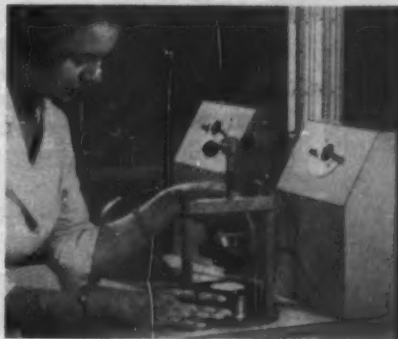
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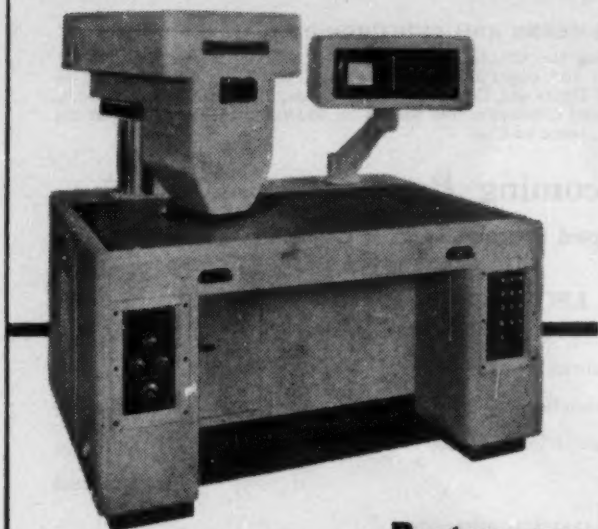
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(Abstracts section only—see January issue, page 2)

NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc., should consult pages 1-8 of the January 1961 and pages 277-285 of the July 1961 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Scouring and Dyeing Blends for the Carpet Trade	R. C. Cheetham
Dyeing and Finishing Terylene-Linen Fabrics	I. E. Haden
The Problem of Illumination in Colour Matching	K. McLaren
Combined Dyeing and Finishing of Cellulosic Piece Goods	I. D. Rattee
An Introduction to Organic Pigments	F. M. Smith

COMMUNICATIONS

Two Problems associated with the Blending of Coloured Fibres	J. C. Guthrie, (Miss) J. Moir and P. H. Oliver
Photochemistry of Sodium Chlorite in Solution, with special reference to Chlorite Bleaching	G. Kujirai and I. Fujita
Sorption of Acids by Wool from Mixtures of the Acids I—Sorption of Hydrochloric Acid and Dichloroacetic Acid or Monochloroacetic Acid	P. Larose and R. Donovan
Paper Chromatography of Insoluble Dyes	J. Sramek
Characterisation of Solvent-soluble Dyes by Optical Transmission Methods	I. F. Trotter

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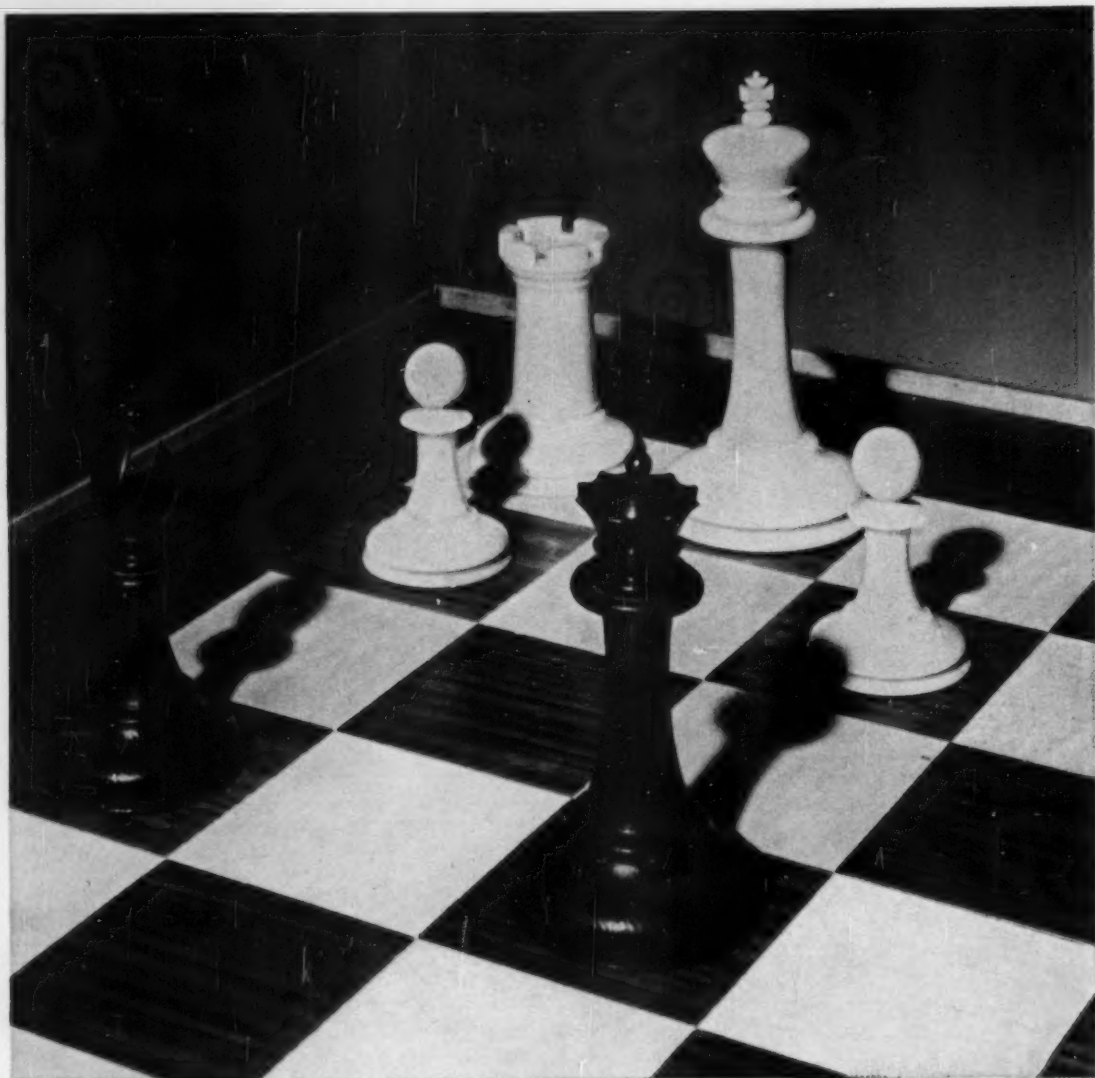
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THE JOURNAL

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Proceedings of the Society

Legal and Practical Problems of Effluent Disposal

J. H. HARWOOD

Meeting of the West of England and South Wales Section held at the County Hotel, Taunton on 9th February 1961, Mr. G. Welford in the chair

The current legal position affecting the disposal of trade effluent in sewers, rivers, and estuaries is reviewed. General types of treatment plant are described, and the use of coagulants and of biological treatment for removing polluting matter from trade wastes is discussed. Examples of the treatment of trade wastes from colour-using industries are given and the approximate cost of treatment is indicated.

Disposal of trade effluent is in fact the disposal of water and, when considered in conjunction with water supply and the use of water, comes into its true perspective and forms the conclusion of a cycle which began with the introduction of water. The water supply position in some parts of the country is serious and water conservation is becoming more and more a national problem.

The Legal Position*

Trade effluents may be disposed of to sewers, streams, or the sea. The discharge of effluents to sewers is governed by the Public Health (Drainage of Trade Premises) Act, 1937 and the Public Health Act of 1961, which came into force on 3rd October 1961. Part V of the latter Act amends the law relating to the discharge of trade effluents into public sewers. Discharges of effluent made prior to the 1937 Act were exempt from its legislation provided that there was no change in nature and composition and no increase in volume or rate. The new Act now controls these discharges and enables local authorities to make charges for the reception and disposal of all trade effluents and to impose conditions on their acceptance. These conditions may relate to the nature or composition of the effluent, its temperature, the elimination of certain specified constituents, the maximum quantity and the highest rate of flow. A charge may be made for the reception and treatment of the effluent at a sewage works.

The local authority can now require that inspection chambers or manholes be provided and maintained to enable samples to be taken, and that records be kept and returns made of volume, rates of discharge, and nature and composition of the

effluent. Pretreatment of the effluent before its discharge to the sewers may be necessary to comply with the conditions imposed. Consents for the discharge of trade effluents may be revised at two-yearly periods. The factory owner has the right of appeal to the Ministry of Housing and Local Government against the imposition of conditions considered to be unreasonable.

The 1961 Act in effect introduces retrospective legislation which, if applied in full by local authorities, would involve factories that have been disposing of their effluents into sewers in considerable expense in providing measuring and recording equipment and sampling points, and in paying for the reception of the effluent.

A research laboratory is now considered to be an industrial establishment and effluents discharged will be subject to control.

The disposal of trade effluents to sewers is the most satisfactory method and is to be recommended wherever feasible, although one drawback to acceptance of trade effluents into local-authority sewers is often lack of capacity in the sewers and at the sewage works, but this should be overcome as new works are constructed.

Disposal of trade effluents to streams, rivers, and the sea is beset by much legislation, which is divided into two sections, common law and statute law. Section 11(6) of the Rivers (Prevention of Pollution) Act, 1951 states that "nothing contained in this Act shall affect the law relating to nuisance"; thus, compliance with statutory provisions is no protection against an action at common law. Pollution, as defined in actions brought under common law, is any act which changes the natural qualities of the water in any river or stream. It is very difficult to comply with the common law position as it stands today, and any person who

* Legislation passed since the lecture was given has been taken into account in this section.

causes pollution does so at the risk of heavy financial damage and curtailment or even closure of operations by court injunction under common law action.

The statutes relating to pollution of rivers and estuaries are—

1. Sea Fishery Regulations, 1888
2. Salmon and Freshwater Fisheries Act, 1923
3. River Boards Act, 1948
4. Water Acts, 1945 and 1949
5. Rivers (Prevention of Pollution) Act, 1951
6. Clean Rivers (Estuaries and Tidal Waters) Act, 1960
7. Rivers (Prevention of Pollution) Act, 1961

The first two statutes prohibit the discharge into sea or fresh water of solid or liquid matter that would be detrimental to fish, to spawning grounds, or to spawn or food of fish. Both these Acts have caused much trouble because of the difficulty of establishing what constituents of certain effluents would in fact be poisonous or injurious to fish in the circumstances of any particular case at any particular time.

The River Boards Act, 1948 set up the River Boards to administer fisheries, river pollution, and land drainage. On the whole they have tackled a difficult task very well. Under Section 9(8), a River Board may legally demand information from a company in relation to its water abstraction and discharge, and under Section 15 power is given to Officers to sample any effluent. The results of any analysis are not admissible in law unless the sample is divided into three parts, each sealed in a container, and one part delivered to the owner of the effluent, one retained for comparison, and one subjected to analysis. Co-operation with the River Board Officers, in particular as regards sampling, is obviously well worth while.

The Rivers (Prevention of Pollution) Acts of 1951 and 1961 are the main controlling statutes. It is an offence under these Acts to pollute a river, and all companies discharging effluents to rivers would be in danger of prosecution by River Boards were it not for two protective provisions. Under Section 2(3) it is a defence to establish (a) that the effluent could not have been discharged otherwise than into the river and (b) that all reasonable and practical steps have been taken to prevent the effluent being unnecessarily noxious, poisonous, or polluting. The minimum amount of treatment means at least the use of balancing tanks, control of pH, and sedimentation of the effluent. The standard of treatment expected is defined in two different ways—

(a) "working standards", which have no legal force but are used by River Boards to classify effluents

(b) "consent standards", conditions imposed on effluents, which have full legal force.

The main purpose of the 1960 Clean Rivers Act is to make it essential for River Board consent to be obtained before a new discharge of effluent is made to an estuary or before a substantial change is made in the nature or volume of an existing discharge. The word "substantial" is not defined in the 1951 Act and has not yet been the subject of a

Court decision. The main consideration attached to any alteration will undoubtedly be its effect on the river or estuary.

A summary of the legal requirements is set out in a useful booklet published by the Federation of British Industries¹. It is easy to understand and forms a useful guide to legislation on the subject, but is now not completely up to date.

There is no doubt that more active steps to prevent pollution of rivers and streams will be taken in the future, and the recent report of the Armer Committee² recommended some ways of improving the legislation which have been incorporated into the 1961 Act. The Act will come into operation in stages commencing on a date yet to be appointed. It extends the 1951 Act by giving River Boards power to control old (pre-1951) discharges to streams, rivers or canals. Under the Act the "consent procedure" applicable to new (post-1951) discharges is extended to cover old discharges, and it will be necessary for factories to apply to the River Boards for permission to continue the discharge. In giving their consent the Boards can lay down conditions with which the effluent must comply, within three months, or after a stipulated period. Once a consent is operative it must at all times be complied with, otherwise an offence is committed. It is, therefore, most important that companies should not accept a condition of consent with which an effluent, after reasonable treatment, cannot comply. The committee recommended no change in the common law relating to river pollution, but a minority report stressed the need for change.

On the whole, the most acceptable solution for industrialists is to have their effluents accepted into the public sewage system wherever possible. Any charge for treatment is almost certain to be considerably less than the costs involved in construction, maintenance, and operation of an individual plant. Once an effluent has been accepted into a sewer, then, unless it can be proved that a major change in composition has occurred, the responsibility for treating and maintaining the quality of the sewage works outfall devolves on the local authority.

Treatment Plants

There are several very useful books dealing with the treatment of trade wastes³. These publications cover the general methods of treatment and types of plant used; specific types of waste waters are dealt with in individual chapters.

The minimum equipment necessary to treat a normal works effluent consists of balancing tanks, and pH adjusting and sedimentation equipment. Where wastes are to be discharged into streams, biological treatment may also be necessary.

Balancing tanks form an essential part of many works systems. The design varies according to individual circumstances, but basically provision must be made for some hours storage to minimise differences in the nature of the effluent and to take account of periodic discharges of some highly concentrated or perhaps acidic waste. Sometimes it may be preferable to isolate different sections of

the wastes and to combine them at a fixed rate. As an example the experience of one works may be quoted: an acid waste was being treated with large quantities of lime every day. However, results were not uniformly satisfactory, for at certain times during the day the effluent became highly alkaline and a yellow, cloudy liquor passed through the settlement tanks to the river. Investigation showed that strongly alkaline wastes from a boiling process were discharged periodically during the day. The sewage system was reconstructed to take these wastes to a separate storage tank from which they were added at a controlled rate to the main effluent. The use of lime was discontinued and a very satisfactory effluent was obtained uniformly throughout the day.

It is, therefore, sometimes possible to combine balanced effluent treatment and pH control. In all instances, careful assessment of individual effluents is very desirable before embarking on a pH control scheme. The formation of floc in sedimentation tanks depends markedly on the pH of the effluent, and for some types of effluent the permissible range of pH may be narrow. The pH is controlled by an acid or acid salt, e.g. sulphuric acid or aluminium sulphate, or an alkali, usually lime.

Arrangements can be made for the addition to be made completely automatically according to the pH of the effluent. In the Kestner patent continuous neutraliser, for example, waste acid is discharged at a predetermined rate by means of a high-efficiency vortex stirrer. The neutralising agent may be fed in by means of a dry chemical feeder or as a solution or slurry. A new principle of treatment is the use of ion-exchange resins. This has been applied to pickling liquor but the process has so far only reached the laboratory stage and is uneconomic⁴. Flue gases which may contain as much as 15% of carbon dioxide are being used increasingly for the neutralisation of alkaline wastes. Sulphur-dye wastes containing up to 30 p.p.m. of sulphides were treated counter-currently with flue gas containing 12–14% of carbon dioxide. The pH of the effluent was adjusted to an equilibrium value of 6.4, and all of the sulphides were removed, presumably by reaction with the sulphur dioxide present in the flue gas, when the retention time was 22 min⁵.

Sedimentation Treatment

In some instances an effluent may be treated by sedimentation without the addition of a chemical reagent; in other cases this addition may be essential. The purposes for which industrial wastes are treated with chemical agents include—

- (a) neutralisation
- (b) precipitation of soluble cations or anions as insoluble compounds (e.g. metals as hydroxides, chromates as barium chromate)
- (c) reduction (e.g. chromate to chromic salt)
- (d) replacement of one metal by another (e.g. copper by iron)
- (e) removal of suspended and colloidal matter by coagulation (e.g. use of aluminium sulphate or ferric sulphate).

It is necessary to consider each problem separately, and it is impossible to postulate general rules, although treatment plants are of similar construction. Several general types may be used.

(1) Use of Solid Reagent

The effluent is brought into contact with excess of solid reagent, e.g. in the removal of copper by scrap iron, reduction of chromate over scrap iron, or the neutralisation of certain acid effluents over lump limestone.

(2) Fill-and-draw Unit

When waste waters are discharged periodically, e.g. during the day-time, or when one section of a factory effluent requires special treatment, this type of unit is often satisfactory. Typical layouts have been described⁶. Two treatment tanks in parallel are normally required. The waste waters are discharged into one tank and, at the end of the required period, the appropriate quantities of chemicals are added, the contents of the tank are gently stirred for a time, and the liquid is allowed to settle overnight. Next day the supernatant liquor is discharged and the second tank used for that day's wastes. The sludge from the first tank is pumped out.

A scheme of this kind has the advantages that—

- (a) Waste liquids of variable flow and composition are mixed before treatment
- (b) Ready control of the amount of reagents added is possible
- (c) If underdosage occurs this can be remedied before the effluent is discharged
- (d) Wastage of reagents by overdosage can be avoided
- (e) Day-to-day alterations in the nature of the effluent can be avoided
- (f) Any offensive gases produced can be fed to a chimney.

The main disadvantage is that comparatively large tanks may be required but, especially for small installations, the fill-and-draw unit is often the most economical and trouble-free in action.

(3) Continuous-flow Units

When a larger volume or a continuous flow of effluent has to be dealt with, the liquor is passed through a mixing tank in which the reagents are added, and then through sedimentation tanks. A typical layout has been given⁶.

The sedimentation tanks generally have to be large enough to give a retention time of two to six hours, depending on the settling characteristics of the floc. Difficulties arise when the nature or rate of flow of the effluent varies considerably during the day. The amount of chemicals added generally has to be adjusted to be adequate for the maximum rate of flow or strength of the liquor, which leads to some wastage of chemicals and production of sludge. The variations in composition are best controlled in balancing and mixing tanks placed before the reagent chamber. If considerable variations in flow rate take place, the installation of an automatic flow recorder arranged to control the addition of reagent will often be worth while.

In treatment by coagulants, encouragement of flocculation by gentle stirring for some minutes before sedimentation occurs is advantageous. Bubbles of gas in the suspended particles are also removed. Increasing the flocculation time above 10 min has been shown⁷ to give no additional advantage but, in comparison with samples of sewage that had not been mechanically flocculated, much more rapid settlement was obtained, making the use of smaller sedimentation tanks possible.

DESIGN OF SEDIMENTATION TANKS

Three general types are in use: horizontal flow, upward flow, and radial flow⁸. The rectangular horizontal-flow tanks are the simplest to construct, but manual labour is required for desludging unless the unit is large enough to justify the introduction of mechanical equipment. There are numerous proprietary forms of upward- and radial-flow tanks in which sludge removal is carried out continuously and mechanically. For equal degrees of removal of suspended matter from a liquid, a shorter retention time is usually required in an upward-flow or radial-flow tank than in a horizontal-flow tank. Particularly in smaller units, the economics favour rectangular tanks, since they require less capital investment and maintenance.

COAGULANTS

Aluminium sulphate is a frequently used coagulant. Dissolving tanks may be made from timber, concrete, or mild steel, and must be lined with rubber, lead, or other impermeable acid-resistant layer. Nori acid-resistant tiles set in Proderite cement are very satisfactory. Sulphuric acid is also best stored in a tank constructed of these materials; the safety precautions to be taken in handling the acid are well known. Lime is generally added to effluents as an aqueous suspension; wooden vats or steel tanks fitted with agitators are suitable, since there is no corrosion problem. Ferric sulphate (or chlorinated copperas) is sometimes used as coagulant but corrodes metals, and excess of iron in the final effluent may cause problems; ferrous sulphate is not as difficult to handle as chlorinated copperas. Organic polymers or polyelectrolytes of high molecular weight have been used successfully. A much smaller amount is required, but the unit cost of these compounds is rather high (about 7s. 6d. per pound). They also tend to be very specific (see Table I).

TABLE I

Specific Action of Polyelectrolyte Coagulants on Effluent from Sand-washing Plant

Type of Sedipur	Suspended solids after 10 min settlement (dosage 0.1 p.p.m.)
LK 4000	150
CD 4	118
PA 4	14
LD 4	72
AP	150
LK 4002	130
Nil	150

An example of the use of polyelectrolytes in economic dosages may be given in the case of a sand-washing effluent. The use of aluminium

sulphate was not satisfactory at any economic dose, but a causticised starch (Powderfloc) was effective at 75 p.p.m. and a polyelectrolyte (Sedipur PA 4) at 0.5 p.p.m. (Table II).

TABLE II

Use of Organic Coagulants in Purification of Effluent from Sand-washing Plant

Coagulant	Dosage (p.p.m.)	Suspended solids (10 min settlement) (p.p.m.)
Nil	—	150
Aluminium sulphate	100	96
Aluminium sulphate	300	78
Aluminium sulphate	50	136
Sulphuric acid	200	
Powderfloc	75	20
Sedipur PA 4	0.25	68
Sedipur PA 4	0.5	20

Various types of starches, e.g. corn and potato starch, are used as coagulant aids. Tragacene is a causticised starch said to be more stable in solution; ordinary starch solutions are unstable. The Unifloc process depends largely on the use of starch as coagulating agent, assisted by mineral salts; very successful results have been obtained, in particular with coal slurries. In certain cases starch can be more effective than inorganic coagulants.

Where the coagulant is added to a section of the plant under pressure, the solution can be charged by means of a variable-stroke ram or rubber-diaphragm pump. For direct addition of the coagulant to water channels, the solution can be run or syphoned from the tank through a measuring device or proportioning pump and thence to the point of application.

Control of pH, considered earlier, is often important in chemical coagulation processes. When aluminium sulphate is used, maintenance of the pH between 6 and 8 gives the most effective results.

Some improvement can be effected in all types of wastes by the combination of pH control and sedimentation, with or without the addition of chemicals. The extraction of suspended solids is often virtually complete, but the concentration of oxidisable, soluble organic matter is not often reduced by more than 50%. There is a strong case for keeping sewage and trade effluents separate if the effluent is to be treated at the factory, in order to avoid contamination of sludge with decomposing organic matter.

SLUDGE DISPOSAL

The sludge collected in the sedimentation process frequently presents a disposal problem. Occasionally there may be convenient old quarries or mine workings into which the liquid sludge may be pumped, but normally it is necessary to de-water the sludge to obtain it sufficiently dry to be handled. The cheapest method, if land is available, is to construct sludge-drying beds into which the liquid can be pumped to dry out gradually. Certain chemicals, e.g. ferric chloride and aluminium chloride, may be added to accelerate drying. Aluminium sulphate and chlorinated copperas may also be

used, but 25–40% more is required. Highly basic aluminium chloride ("chlorohydrate") gives improved sludge-bed drying rates. Where the amount of sludge to be handled is large, installation of rotary vacuum filters may be justified.

Biological Treatment

A biological filter consists essentially of a concrete base, under-drains, a peripheral wall, the filtering medium, and a distributor. The base slopes to a drainage point. The distributor is a rotating nozzle system driven by reaction jets. The filtering medium consists of tile supports, large stone, 3–6 in. in diameter, and then the actual medium, stone, slag, cinder, or coke of $\frac{1}{4}$ –2 in. diameter⁹.

The medium becomes covered with a film of bacteria, fungi, and other organisms, and it is these that bring about purification of the liquor. The filter also contains large amounts of worms, fly larvae, and other microscopic animals which feed on the biological film and prevent ponding.

Many trade wastes contain substances such as cyanides, phenols, and formaldehyde, all of which can be toxic to bacteria and yet, surprisingly, many of these wastes are proving amenable to treatment by aerobic biological methods. A biological filtration plant at one factory is reported¹⁰ to be achieving about 95% reduction in phenols and in permanganate value. Considerable success has also been achieved in the treatment of cyanides on percolating filters¹¹. Cyanide is converted to ammonia and provides the only source of carbon and nitrogen to the micro-organisms bringing about the conversion.

Biochemical reduction processes are useful for the treatment of trade wastes when the concentration of organic matter is high and when the substances to be removed are non-oxidisable but capable of reduction to harmless products. Many dyes are decomposed by anaerobic treatment and successful results have also been reported for slaughter-house wastes and cellulosic wastes from the manufacture of hardboard.

A valuable discussion of the factors affecting the treatment of industrial wastes by biological oxidation and anaerobic digestion has been given¹². The factors include availability of organic food and oxygen, types and number of micro-organisms, pH, and temperature. The waste waters dealt with include those from the manufacture of textiles, gums and antibiotics, and from beet sugar factories, canneries, dairies, fermentation industries, packing houses, pulp and paper mills, and chemical works, particularly wastes containing phenol and formaldehyde.

Specific Applications

So far this paper has been concerned with the general legal background and methods of treatment of trade effluents. Information on the treatment of wastes from the colour-using industries will now be given.

PAPER-MILL WASTES

The addition of coagulants to paper-mill wastes before sedimentation is well established as one of

the most satisfactory methods of treatment. In typical cases almost complete removal of suspended and polluting matter has been achieved. Normal paper-mill waste is acid, that from mills treating straw rope is alkaline. The pH of the effluent should be adjusted to about 7 to obtain the most satisfactory results. A mixture of sodium aluminate and aluminium sulphate has been very successful in certain instances, yielding an excellent effluent¹³.

TANNERY EFFLUENTS

These generally prove to be very obnoxious effluents of variable composition. Experience has shown that considerable economy in the use of chemicals can often be achieved by ensuring complete mixing of the effluents from different sections of the process before treatment. Some sections discharge a very alkaline liquor containing lime, sodium sulphide, etc., while from other parts chromic acid and acid vegetable tannage, giving an acidic liquor, is discharged. Provision of adequate storage facilities often effects a considerable economy.

About half of the high oxygen demand, as measured by the oxygen absorbed (O.A.) test, in a tannery effluent is due to materials in solution which cannot be removed by coagulation. Sometimes the effluent is best kept neutral, but in other cases much higher pHs give satisfactory results; this effect is presumably controlled by the proportion of other chemicals in the effluent. Increasing the dosage of the coagulant does not materially affect the oxygen demand of the effluent. With a raw effluent of O.A. 2300 p.p.m., 60 p.p.m. of aluminium sulphate reduced the O.A. to 1500 p.p.m.; 500 p.p.m. gave a further reduction only to 1240 p.p.m.

In general, unless the effluent is discharged to a domestic sewer, coagulation by itself is not sufficient. Chemical precipitation can generally reduce the load on biological filters by one half, but these effluents are so complex that separate investigation of each is necessary to determine the optimum conditions for treatment¹⁴.

DYEHOUSE AND TEXTILE WASTES

These also are very complicated effluents to deal with, and a detailed investigation is necessary in each case. In addition to various types of dye, sulphur-containing compounds, bleach residues, and highly alkaline or acid liquors may be discharged. Where several different types of treatment are carried out on the same site, adequate mixing is essential before treatment is attempted. With sulphide-containing liquors, the addition of certain coagulants leads to the liberation of hydrogen sulphide. If this can successfully be vented to the atmosphere through a chimney without causing a nuisance, the method of treatment may prove very useful, since liquors containing sulphides cannot be discharged into a stream or sewer.

Many highly coloured dye wastes are successfully treated and the colour is often reduced or completely removed by the formation of aluminium lakes. The best pH for treatment depends on the composition of the effluent, but in many cases

TABLE III
Treatment of Dyehouse Effluent

	pH	Floc	O.A. (p.p.m.)	Suspended solids (p.p.m.)	Approx. cost of chemicals (d./1000 gal.)
Blank	11.0	Poor	116	32	—
350 p.p.m. H_2SO_4 + 180 p.p.m. aluminium sulphate	6.5	Good	100	92	0.45
400 p.p.m. H_2SO_4 + 180 p.p.m. aluminium sulphate	6.0	Very good	98	32	0.47
650 p.p.m. aluminium sulphate	8.0	Very good	83	32	1.07

TABLE IV
Coagulation Treatment of Dyehouse and Textile Wastes

	Addition	pH	O.A. (p.p.m.)	Suspended solids (p.p.m.)	Approx. cost of chemicals (d./1000 gal.)
Dyehouse, bleaching and finishing works	nil (settled)	10.0	53	80	—
	400 p.p.m. aluminium sulphate	7.0	38	Nil	0.66
	100 p.p.m. aluminium sulphate + 260 p.p.m. sulphuric acid	4.0	45	22	0.28
Mixed dyehouse and wool-scouring effluent	nil (settled)	7.5	96	28	—
	100 p.p.m. aluminium sulphate + 50 p.p.m. lime	6.0	91	4	0.18
Highly coloured dyehouse waste containing sulphur and vat dyes, bleach, and finishing liquors	nil (settled)	7.8	107	44	—
	200 p.p.m. aluminium sulphate	6.1	70	8	0.72
	100 p.p.m. hypochlorite required to discharge residual bright green colour to give water-white effluent				
Blue-grey opaque suspension from dyehouse and scouring plant	nil (settled)	7.7	364	500	—
	1000 p.p.m. aluminium sulphate	6.0	69	32	1.6
Dyehouse effluent	nil (settled)	10.4	87	120	—
	600 p.p.m. aluminium sulphate	7.4	35	32	1.0

considerable economies in the amount of coagulant needed can be effected by using sulphuric acid to make the effluent nearly neutral.

Table III shows that an additional 50 p.p.m. of sulphuric acid reduced the amount of suspended solids from 92 to 32 p.p.m., and the use of 400 p.p.m. of sulphuric acid reduced the quantity of aluminium sulphate needed from 650 to 180 p.p.m. A greater reduction in O.A. cannot be obtained since much of the demand is caused by materials in solution.

Similar results can be quoted for a number of different effluents to indicate the improvement that may be effected. In Table IV only the dosages of chemicals giving the optimum results for the effluent are given. As an indication of the cost of treatment, the approximate cost of chemicals for treating 1000 gallons of effluent is quoted in each instance.

These results show that a treated liquor acceptable in local-authority sewers can normally be obtained by coagulation, but the residual O.A. is usually still high. Soluble dyes can frequently be removed by treatment with sodium hypochlorite when the liquor is to be discharged to a river; this renders the discharge less obnoxious in appearance. A considerable amount of information is available on methods of treatment¹⁵.

LAUNDRY WASTES

Effective separation of fats and greases and 75–85% of the polluting matter is obtained by combined coagulation and acid treatment. McCarthy¹⁶ quotes a saving of 32% in the cost of

treatment by prior neutralisation. The sludges obtained contain 41–44% of fats, and the possibility of recovering these has been suggested¹⁷.

Southgate¹⁸ considers that the chemical treatment of laundry wastes is well established. A batchwise system in which a day's effluent is neutralised to pH 6.0 to 6.5 and then treated with coagulant is often the most efficient and economical. Boyer¹⁹ recommends thorough mixing of the chemicals for at least 30 min before settlement, to prevent formation of floating scum. Often an effluent suitable for direct discharge to streams can be obtained²⁰.

Conclusion

There is little doubt that control over effluents will be tightened up in the next few years. The cost of complying with the law will have to be borne by industry, and should be kept in mind as a charge to be anticipated. In most cases acceptance into local-authority sewers, with or without pre-treatment, will probably prove to be the most economical and trouble-free course. Where effluents are to be discharged to streams the standard of treatment demanded will increase, and existing plant will eventually have to be improved or new plant installed. A considerable amount of background experience has now been accumulated, and it may be some consolation to industrialists to know that satisfactory processes have now been worked out for treatment of most trade effluents.

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COMMUNICATIONS

The Cross-linking of Cellulose and its Derivatives I—The Use of Unsaturated Esters in Cross-linking Reactions

W. M. CORBETT and J. E. MCKAY

The mechanism of the cross-linking of unsaturated ethers and esters of cellulose is discussed. An examination by paper chromatography of the acidic hydrolytic products of crotonate esters of cellulose cross-linked by peroxides has shown that many complex reactions occur. As this leads to uncontrolled cross-linking, the possibility of control by the addition of amines to cellulose crotonates is discussed.

It is well established that cross-linking of polymeric material leads to the production of new products with greatly different physical properties. Generally, cross-linking is achieved by treating non-cross-linked polymer with compounds of a multifunctional nature, or by polymerising monomer in presence of a cross-linking agent, although the use of high-energy radiation is increasing in importance. For cellulosic materials, particularly textiles, the deposition of resins, e.g. urea-formaldehyde condensates, has been used extensively to alter both physical and mechanical properties. Such a process is the basis of the crease-resist treatment of fabrics. The reactions which occur during this process are numerous and it has not been possible to ascertain which are responsible for modifying the properties. Because most processes require the use of multifunctional compounds (e.g. formaldehyde), some workers believe that cross-linking is at least partly responsible for the improvement in properties. So far the importance of cross-linking has not been established, and it was because of this that the present work was initiated.

Although many types of reagent are claimed to cross-link cellulose because they insolubilise it, the formation of cross-links has not been proved conclusively in any one case. Of the many reactions described, only a few produce cross-links of a definite nature. For example, formaldehyde, the

simplest reagent, can react in three ways to produce (a) a polyoxymethylene resin not bound to the cellulose, (b) a polyoxymethylene graft to the cellulose, and (c) cross-links consisting of varying numbers of oxymethylene groups. In a study of the effect of cross-linking on properties, attention must be turned towards more definite methods, especially those that facilitate the measurement of the degree of cross-linking by analysis. This is necessary since at present it is invalid to assume a definite relationship between degree of cross-linking and any physical property for an amorphous-crystalline polymer.

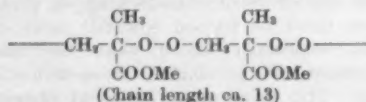
Cross-linking of Unsaturated Esters of Cellulose by Action of Oxygen and Peroxide Catalysts

The conversion of unsaturated derivatives of cellulose to brittle, insoluble materials, often resistant to thermal softening, has been examined by many workers over the last thirty years without either a commercially successful cellulosic product or a detailed explanation of the chemistry of the conversion processes being produced. Most of the investigations have been activated by commercial interests, and have not, therefore, been primarily concerned with the fundamental chemistry of the processes, so that the more recent worker falls heir to a very meagre inheritance of data.

The available information shows that oxygen, organic peroxides, and ultraviolet radiation are effective agents in "polymerising" both unsaturated ethers¹ and esters² of cellulose. It is reasonable to suppose that cross-linking of the polysaccharide chains is one of the reactions occurring and may be of prime importance in modifying the physical characteristics of the materials. A review of the observed behaviour of cellulose derivatives toward the above reagents must separate reactions in presence of, and those in absence of, oxygen if the mechanism is to be elucidated.

REACTIONS IN PRESENCE OF OXYGEN

Methacryloyl^{2a}, crotonoyl², and sorboyl^{2a} derivatives of cellulose are slowly rendered insoluble on exposure to air or oxygen. This process is accelerated by heat, ultraviolet radiation, and peroxides. Solutions of the derivatives in organic solvents are converted to insoluble gels without intermediate changes in viscosity, i.e. no soluble cross-linked material is formed^{2b,3}. These treatments can be applied to methacryloyl, acryloyl, and crotonoyl esters of simple sugars and their derivatives⁴ to produce hard glasses and, since the extent of reaction is probably greater in the insolubilisation of the smaller molecules, these systems are more likely to indicate the chemistry involved. Yanovsky and Treadway^{4a} found that glucose pentamethacrylate became insoluble in organic solvents on being heated in air. During this process the carbon content fell from 59.8% to 54.2%, suggesting that combination with oxygen had occurred. Barnes *et al.*⁵, investigating the role of oxygen in vinyl polymerisation, found evidence for the formation of polymeric peroxides in the autoxidation of methyl methacrylate.

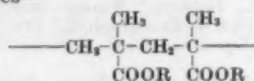


These polymers may be regarded as partial oxidation products, giving finally methyl pyruvate and formaldehyde. Such a copolymerisation process with oxygen would be compatible with the observations of Yanovsky and Treadway and would be accelerated by the action of peroxides and ultraviolet radiation through the formation of free radicals. Polyperoxides are characteristic of conjugated systems and would be expected to be formed also in the cases of crotonates, sorbates, and acrylates. The rates of polymerisation would be dependent upon the ease with which the initial radical production occurred in the various ester monomers. Methacryloyl derivatives are observed to polymerise more readily than acryloyl or crotonoyl compounds^{4a,b}.

REACTIONS IN ABSENCE OF OXYGEN

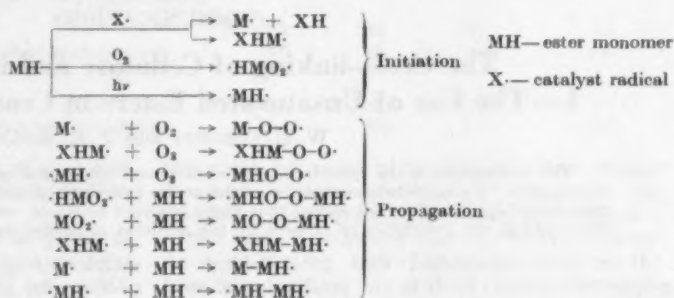
Similar polymerisation effects are observed with polysaccharide and simple sugar derivatives of

methacrylic, acrylic, crotonic, and sorbic acids if these are heated with organic peroxides in nitrogen, but the time required for polymer formation is somewhat longer than in presence of oxygen^{2,4}. In these reactions normal vinyl polymerisation might be expected to operate; thus, in the case of methacrylates—



FACTORS COMMON TO BOTH REACTION TYPES

Both in presence and in absence of oxygen, the mode of initiation will depend upon the character of the unsaturated group and of the catalyst. The initial formation of a free radical from the monomeric molecule can be accomplished either by the addition of a catalyst radical to the double bond or by the abstraction of a hydrogen atom, and it is possible that both processes may act concurrently. The course of subsequent reaction of the monomeric radical will be directed by the availability of further monomers for the growth of a polymer chain, and by the free radicals in the system for the frequency and nature of chain termination.



Termination of polymerisation will occur by the reaction of the above radicals with any radicals present.

REACTIONS OF CROTONOYL DERIVATIVES OF CELLULOSE

Crotonoyl esters were selected for study because of their relative stability to incidental reaction during preparation and purification. The initial object of the work was to examine the acids liberated by the hydrolysis of peroxide-treated cellulose derivatives, with a view to estimating the amount of inter-ester combination that had occurred. From the above diagrammatic representation of the various reactions it will be seen that polycarboxylic acids will arise from inter-ester reactions, whereas all other reactions will produce monocarboxylic acids. Thus the amount of polycarboxylic acids should provide a measure of the combination of two or more ester groups.

No attempt was made at this stage to avoid intramolecular reaction, since the chemistry of inter- and intra-molecular bridge formations is identical and large extents of reaction would be expedient in developing analytical methods.

Films cast from solutions of cellulose esters of more than 40% crotonoyl content were rendered

TABLE I
*R_{cro}** Values of Acids from Various Crotonates Treated with Benzoyl Peroxide

Cellulose acetate-crotonate (acetyl 35.8%, crotonoyl 18.4%)	Cellulose acetate-crotonate (acetyl 10.0%, crotonoyl 44.1%)	Cellulose tricrotonate (crotonoyl 51.5%)	Lactose octa-crotonate	Secondary cellulose acetate
0-0.2	0-0.2	0-10	0-0.2	—
0.33†	0.36†	0.26	0.33†	—
	0.53	0.45†		
	0.64	0.65		0.70
0.77	0.79	0.80	0.77	0.80
1.00	0.99	1.00	1.00	1.00
1.15	1.12	1.12	1.15	1.15
Ammonium salts of standard acids				
Adipate	Formate	Acetate	Benzoate	
0.25	0.75	0.80	1.15	

* *R_{cro}*—extent of movement relative to that of cellulose crotonate.

† Oxidised by permanganate.

insoluble by heating in air at 100–120°C for several hours. The treatment produced no measurable decrease in the volatile unsaturated acid yield obtained from the hydrolysates, nor any apparent change in the infrared spectra of the films. Ultra-violet irradiation in air also rendered the esters insoluble, but appeared to produce considerable degradation and, since cellulose acetate is known to lose acetyl groups on irradiation⁶, changes in crotonoyl content could not be regarded as meaningful under these conditions.

The gelation of solutions of cellulose crotonates was carried out in order to obtain larger and therefore more easily detectable amounts of reaction. Ester solutions of 1–2% in dioxan formed gels within thirty minutes when heated at 100°C with 1% of the ester weight of dibenzoyl peroxide, over the range of crotonoyl content 8–52%. The highly swollen gels were usually soluble in boiling formic acid, and hydrolysis was completed in 2-N sulphuric acid. When the gels were resistant to formolysis, e.g. those of the tricrotonates, the finely divided, washed, and desolvated gel was dissolved in concentrated sulphuric acid and the solution, after dilution with water to 2 N, was refluxed for several hours. The organic acids were extracted from the hydrolysates with ether and the volatile acid components were removed by repeated steam-distillation. The ammonium salts of the non-volatile components were then examined on paper chromatograms in *n*-propanol–water (70:30). The chromatographic mobilities of the components of the mixtures obtained from various sources, including a control experiment with secondary cellulose acetate, which is not gelled by the peroxide, are listed in Table I.

The mixtures contained a variety of acids in addition to benzoic acid (from hydrogen capture by benzoyloxy radicals), each including at least one component oxidisable by permanganate. The control experiment with secondary cellulose acetate demonstrated the existence of acid-forming reactions other than those involving crotonoyl groups. The acids formed from the acetate are most likely to have arisen from interaction with the solvent, as illustrated by the acids detected on

decomposing dibenzoyl peroxide in dioxan at 100°C (Table II). Since the yield of non-volatile acids is of the order of 0.5% by weight of the original ester, it is clear that trace side-reactions may contribute to the acid mixtures isolated. The formolysis stage in degrading the gels is a likely source of artefacts, and the non-volatile acids detected on decomposing dibenzoyl peroxide (1%) in a 5% solution of crotonic acid in formic acid are also given in Table II. Benzoyl peroxide in formic acid alone, however, does not give rise to any detectable amounts of non-volatile acids.

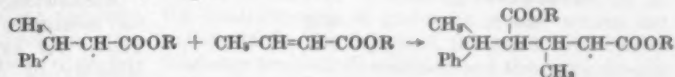
TABLE II
R_{cro} Values of Acids from Dibenzoyl Peroxide in Dioxan and in Crotonic Acid-Formic Acid Mixture

Bz ₂ O ₂ (1%) in dioxan	Bz ₂ O ₂ (1%) in crotonic acid-formic acid mixture
0.70	0.11
1.05	0.33*
1.19	0.84*
	1.16*

* Major components.

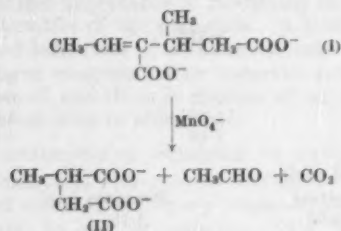
It must be supposed that very little peroxide will be left undecomposed at this stage in the treatment of a gel, but a significant contribution of acids may still occur from side-reactions during formolysis.

From the various observations, one can only conclude that several reactions operate, involving the crotonoyl substituents. These presumably include direct addition, initiated by benzoyloxy or phenyl radicals, to yield non-oxidisable bridges, e.g.

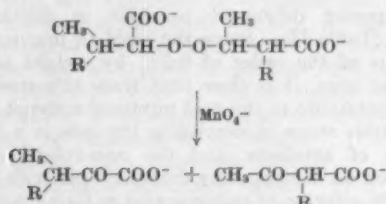


Some evidence for the combination of catalyst decomposition products was obtained from experiments with bis-*p*-bromobenzoyl peroxide which indicated the presence of ca. 1% of combined bromine in the ester gels. At least one oxidisable combination is formed by some process other than addition. This might be an unsaturated molecule, formed by a mechanism involving hydrogen abstraction, or a peroxide structure. The component with *R_{cro}* = 0.45 from the hydrolysate of a

tricrotonate gel compares in chromatographic mobility with α -ethylidene- β -methylglutarate (I) and, on oxidation with permanganate, yields a product with $R_{cro} = 0.22$, which is the mobility of α -methylsuccinate (II), the oxidation product of α -ethylidene- β -methylglutarate.



A peroxide structure could conceivably react in a similar manner, e.g.



The unknown nature of R makes the interpretation of chromatographic observations exceedingly uncertain. The oxidation product of the unknown salt did not show a reaction with 2,4-dinitrophenylhydrazine on chromatograms (orange spots on a yellow background with laevulinate and pyruvate), but the test is probably not highly sensitive.

REACTIONS WITH *t*-BUTYL PEROXIDE

The gelation of the esters with *t*-butyl peroxide in dioxan was examined in the hope that the acid mixtures isolated from the hydrolysates might be less complex than those found in the reactions catalysed by benzoyl peroxide. The reactions were carried out at 120°C so that methyl radicals would predominate. However, the products detected proved to be no less complex than in the previous experiments (Table III).

TABLE III

R_{cro} Values of Acids from Cellulose Acetate-crotonate and Methyl Crotonate Treated with *t*-Butyl Peroxide

Acetate-crotonate (acetyl 10.0%, crotonyl 44.1%)	Methyl crotonate
0-0.2	0.13
0.42*	0.44
0.54	
0.66	
0.78	0.77
1.00	0.98
1.09	1.18

* Oxidised by permanganate.

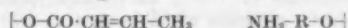
Cross-linking of Unsaturated Esters of Cellulose by Addition of Amines

From these exploratory examinations it is clear that, although peroxide-catalysed modification of unsaturated esters of cellulose may be readily

achieved, the operative processes are exceedingly complex and thereby unsuitable for a quantitative study of cross-linking. It is possible that suitable peroxide-catalysed, or at least free-radical, systems could be devised to be of practicable simplicity for this purpose. Nevertheless, it seems expedient to obtain the simplest system possible, including a single type of reaction product, the absence of polymeric bridge formation, and the possession of easily identifiable and determinable features from the analytical viewpoint.

Attention has therefore been turned to the cross-linking of cellulose crotonate by the addition of diamines, as reported by Engelmann and Exner². This system provides not only a simple means of cross-linking, but also a simple method of analysis. Thus, saponification of a treated film will yield a dibasic amino acid for every cross-link, and a monobasic amino acid for every diamine molecule that has undergone partial reaction through one amine group only. Separation of the two acids, e.g. by paper chromatography, followed by their estimation, facilitated by the use of radio-isotopically labelled diamine, should provide a fairly accurate and simple method of determining the degree of cross-linking.

Although the above system is excellent in many respects, it suffers from the disadvantage that both inter- and intra-molecular cross-links will be produced, and the method of analysis will estimate all cross-links. It may be possible to overcome this difficulty by modification of the system by treating cellulose crotonate with an aminocellulose, e.g.



Such a system can only produce intermolecular cross-links. Further, the use of radio-isotopically labelled crotonyl groups should provide a simple method of estimating cross-links, for saponification of cross-linked material will liberate any unchanged crotonyl groups, but crotonyl groups involved in cross-linking will remain attached to the cellulose by virtue of the amine link. Thus, determination of the residual activity of the saponified film will give a direct measure of intermolecular cross-linking.

In subsequent papers we report progress towards the application of these and other reactions to the controlled, unambiguous cross-linking of cellulose and its derivatives.

* * *

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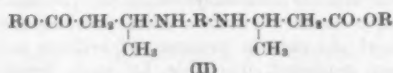
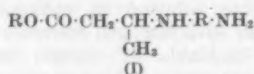
The Cross-linking of Cellulose and its Derivatives

VI*—The Addition of Amines to Cellulose Crotonates

J. E. McKAY and W. TAYLOR

The reactions of cellulose-acetate crotonate mixed esters with ethylenediamine, ammonia, and *n*-hexylamine have been studied. In all cases, addition of the amine to the double bond occurs readily, but only the first two give insoluble, and therefore cross-linked, products. Aminolysis also occurs, but this only becomes of importance after 50–100 hours. Sodium hydroxide hydrolyses the cross-linked films and the products have been identified as far as possible by paper chromatography.

The purpose of these investigations and the reasons for selecting the present system have been discussed in Part I of this series¹. In the general case of addition of a diamine to a crotonate, in such a way that the former is in large excess, the principal product will be that of mono-addition, viz. the appropriate aminoalkylaminobutyric ester (I); the di-addition product, the *NN'*-alkylenedi(amino-butyrate) (II), will constitute a relatively small fraction, the size of which will depend on the comparative rates of addition and the stabilities of the products.



Experiments with model compounds of low molecular weight have shown that these products are obtained not only from simple aliphatic esters but also from sugar derivatives², and thereby that the reaction may be justifiably applied to cellulosic systems. This communication describes some of the preliminary results of the addition of amines to cellulose acetate-crotonate mixed esters.

Addition of Ethylenediamine

A series of cellulose acetate-crotonate mixed esters, ranging from virtually triacetate through increasing crotonoyl content to tricrotonate, was prepared by treating cotton with mixtures of acetic and crotonic anhydrides in dichloromethane in presence of small amounts of perchloric acid. Extensive depolymerisation occurred during the acylations, which yielded products of D.P. 100–200. Thin films of these esters were obtained by allowing 1–2% chloroform solutions to evaporate on mercury surfaces. The films thus obtained were immersed in 5% solutions of ethylenediamine in the desired solvent at room temperature, removed after the

appropriate reaction time, washed with ethanol, continuously extracted with ethanol containing oxalic acid in a Soxhlet apparatus, and dried *in vacuo* over phosphorus pentoxide at 60°C before being analysed.

VARIATION OF COMBINED NITROGEN WITH CROTONOYL CONTENT

The nitrogen contents measured after treatment of a series of esters for 24 h in 5% ethylenediamine in ethanol-chloroform (9:1 v/v) are shown in Table I. N/N_{max} represents the observed nitrogen content as a percentage of that required for complete reaction of the crotonoyl groups to give mono-addition products, and N/N_{min} a similar comparison on the basis of complete di-addition.

TABLE I

Ester	Crotonoyl content (%)	Acetyl content (%)	N found (%)	N/N_{max}	N/N_{min}
1	9.2	40.0	0.82	23.7	45.5
2	24.5	21.8	1.50	18.6	33.3
3	36.5	14.6	2.10	18.5	33.0
4	44.1	10.0	1.50	11.6	20.0
5	47.5	4.3	1.20	8.0	15.1
6	51.5	0	1.80	12.5	21.2
7	0	43.7	0.06	—	—

The general trend of the results indicates a decline in the efficiency of combination with increasing crotonoyl content. It could be considered that, as the concentration of crotonate groups within the volume of the film increases, the greater will be the tendency towards di-addition and the more closely is the extent of reaction represented by N/N_{min} . At the same time it is necessary to bear in mind that the increasing crotonoyl content must also affect those properties of the film, e.g. the degree of order and the extent of swelling, upon which the penetration of the reagent is dependent. Since samples 1–6 became completely insoluble in organic solvents, it seems likely that some cross-links were formed.

* Part V—Corbett, McKay, and Taylor, *J. Chem. Soc.*, in the press.

VARIATION OF COMBINED NITROGEN WITH TIME

The effect of extending the time of reaction was next examined. Table II shows the change in nitrogen content observed when ester 3 was treated in 5% ethylenediamine in ethanol-chloroform solution at room temperature for 100 h.

TABLE II

Time of treatment (h)	N found (%)	N/N _{max}	N/N _{min}
1	0.38	3.4	5.9
2	0.83	7.4	13.0
3	0.81	7.2	12.7
4	1.07	9.6	16.7
6	1.21	10.8	18.9
24	2.01	17.9	31.5
30	2.18	19.4	34.0
48	2.54	22.6	39.5
100	3.13	27.9	49.0

Although the samples became insoluble in organic solvents after 2 h, the nitrogen content continued to increase throughout the period of reaction. In order to obtain maximum efficiency of reaction, films were transferred after 6-h treatment to a 5% ethylenediamine solution in chloroform, in which a high degree of swelling was known to occur. Table III shows the results obtained with esters

TABLE III
Addition of Ethylenediamine to Cellulose Acetate-crotonates in a Swelling Medium

	Time of treatment (h)	N found (%)	N/N _{max}	N/N _{min}
Ester 1	0	0.75	21.5	41.7
	6	0.87	24.8	48.4
	24	1.20	34.5	66.5
	30	1.28	36.6	71.3
	42	1.60	47.3	90.5
	102	1.41	40.8	78.5
Ester 3	160	0.30	8.6	16.7
	0	1.13	10.1	17.7
	18	2.56	22.7	40.1
	24	3.18	28.4	49.7
	42	3.99	35.6	62.1
	138	2.17	19.4	33.9

TABLE IV
Variation of Ester Content in Reaction of Ethylenediamine with Cellulose Acetate and Acetate-crotonate

	Time of treatment (h)	Acetyl content (%)	Ester content (m equiv/100 mg)
Triacetate	0	45.4	1.06
	16	44.6	1.04
	24	43.7	1.02
	45	42.9	1.00
	70	42.8	1.00
	140	39.1	0.91
	170	37.3	0.87
		Nitrogen content (%)	
Ester 1	0	0.75	1.04
	24	1.20	0.96
	102	1.41	0.94
	160	0.30	0.34

1 and 3 in the chloroform medium. Samples of ester 1 taken after 24, 102, and 160 h were analysed for combined volatile acids, and the values corrected from the nitrogen contents to give the total combined acids. A comparison was made with the fall in acetyl content observed for cellulose triacetate treated with 5% ethylenediamine in ethanol-chloroform (1:1 v/v) at room temperature (Table IV).

Although the addition proceeded much more rapidly in the chloroform medium, aminolysis accelerated enormously during the later stages of the treatment, with the loss of substantial amounts of ester and nitrogen. From the behaviour of ester 1 it is apparent that the initial rate of aminolysis is slow and comparable with that of the triacetate, but as the addition proceeds aminolysis is accelerated. This can be appreciated to the extent that the aminobutyl groups will be much more susceptible to aminolysis than the acetyl or crotonoyl groups, but the enormous drop in ester content would indicate that the cleavage of acetyl groups must also become accelerated. It is obvious, however, that extensive aminolysis can be avoided by using moderate times of reaction.

In order to produce a crotonoyl derivative which contained a small number of crotonoyl groups introduced in a random manner and which was of sufficiently high D.P. to possess good film-forming properties, cellulose triacetate was treated with crotonic anhydride in dichloromethane in presence of magnesium perchlorate. A small amount of ester exchange occurred, yielding a product of ca. 1% crotonoyl content. Treatment with 5% ethylenediamine solution in ethanol-chloroform produced films with nitrogen contents of ca. 0.1% which were insoluble in organic solvents. An ester of similar crotonoyl content, produced by treating secondary cellulose acetate in acetone with crotonoyl chloride in presence of sodium acetate, was not rendered insoluble by such treatment. The specific viscosity of this material was reduced to approximately half of the original value by the amine treatment, but since that of cellulose triacetate was unaffected by a similar treatment, it can only be presumed that the change is a result of combination with the amine rather than of degradation by it.

Addition of n-Hexylamine

It could be argued that the insolubilisation of these films is due to the formation of nitrogen-bearing side-chains rather than to the formation of intermolecular bridges. To test this possibility, films of ester 5 were treated with 10% n-hexylamine in ethanol-chloroform (9:1). The observed combination with nitrogen is shown in Table V.

TABLE V
Addition of n-Hexylamine to Cellulose Acetate-crotonate

Time of treatment (h)	N found (%)	N/N _{theor} (%)	Ester content (m equiv/100 mg)
6	0.38	3.4	0.90
75	1.02	9.0	0.56
100	1.15	10.2	0.61
130	Film soluble in ethanol		

Although the addition proceeded up to some 10% of the theoretical value, the films did not become insoluble in chloroform but, on the contrary, eventually became soluble in ethanol. The figures in the final column indicate that considerable aminolysis also occurs in this system.

Addition of Ammonia

Since ammonia is capable of forming di-addition compounds with simple esters of crotonic acid³, it should also be capable of introducing additive cross-linkages into cellulose crotonates. Table VI shows the variation in nitrogen content observed when ester 4 was treated with 5% ethanolic ammonia solution at room temperature. Samples became insoluble after 55-h treatment, but they could be insolubilised after only 6 h by heating. The addition of ammonia is a rather slower process than that of a primary amine.

TABLE VI
Addition of Ammonia to Cellulose
Acetate-crotonate

Time of treatment (h)	N found (%)	N/N _{max}	N/N _{min}
6	0.19	2.3	4.4
24	0.39	4.8	9.1
30	0.34	4.2	7.9
55	0.86	10.3	20.1
90	0.75	9.3	17.2
120	0.71	8.6	16.5

Saponification of Cross-linked Films

Insolubilised films were immersed in 1-N alkali at room temperature for varying times, washed rigorously with water, dried, and analysed. The decreases in the combined nitrogen content are recorded in Table VII.

The bulk of the nitrogen is removed by this method, but analytical methods of greater sensitivity are required to determine the efficiency of the final stages of the saponification.

Paper-chromatographic analysis of the hydrolysate of ester 3 cross-linked by ammonia

TABLE VII
Saponification of Cross-linked Cellulose
Acetate-crotonates

	Time of treatment (h)	N found (%)	N/N _{max}	N/N _{min}
Ester 1 cross-linked by ethylenediamine	0	0.97	28.7	56.5
	15	0.04	1.2	2.4
	24	0.05	1.5	2.9
	39	0.02	0.6	1.2
Ester 4 cross-linked by ethylenediamine	0	2.50	19.4	33.0
	60	0.14	1.1	1.9
Ester 2 cross-linked by ammonia	0	1.80	41.9	78.5
	16	0.22	5.1	9.6
Ester 3 cross-linked by ammonia	0	0.72	10.4	20.0
	24	0.11	1.6	3.1

showed the presence of two components corresponding in mobility to β -aminobutyric acid and $\beta\beta$ -iminodibutyric acid. In the cases where ethylenediamine was the cross-linking agent, only β -2'-aminoethylaminobutyric acid was detected, but this is due in some measure to the difficulty of obtaining a sufficiently sensitive reagent for the detection of NN' -ethylenedi(β -aminobutyric acid) on paper chromatograms.

* * *

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Further Tests for Colour Vision

F. JORDINSON

The difficulties of diagnosing defective colour vision are discussed, and combinations of different kinds of tests suitable for diagnosis are suggested. A detailed examination of the colour vision of key employees in a commission dyehouse is described, and an attempt is made to interpret the results in terms of deviations from normal colour vision and to assess the relative degree of colour discrimination.

Colour-vision Tests

In a very interesting lecture delivered to a medical audience, Wright¹ has discussed the problem of diagnosing defective colour vision. He suggests that the first stage is to establish the capacity to discriminate colours, then to ascertain the colours that are confused. Yet it is difficult to design tests to give the above answers, chiefly because in such tests there are no standard conditions for viewing, and several factors may influence the response of the subject, e.g. light sensitivity, adaptation, visual acuity, and even his temperament, state of health, and his reaction to the examiner. To these uncertainties may be added the effect of macular pigmentation and yellowing of the lens of the eye, both of which may affect the quality of the light stimulus reaching the retina.

The present author² has given examples of modern colour-vision tests classified as—

- (1) confusion-chart tests or pseudoisochromatic charts, in which the examinee is light-adapted,
- (2) lantern tests, where he is dark-adapted,
- (3) matching tests, in which coloured discs have to be arranged in the correct hue sequence.

To these may be added the use of physical instruments, e.g. a tristimulus colorimeter or an anomaloscope, in which a coloured light of known wavelength is matched by means of mixed lights of another quality.

Each of these types of test requires a different set of experimental conditions for testing colour vision, and it is therefore not surprising that results are frequently obtained which are apparently conflicting. This illustrates the great difficulty of laying down an absolute standard for grading the ability either to identify a given colour or to discriminate between two colours. Obviously, most reliance must be placed on the test that corresponds most closely to the manner in which the person to be tested requires to exercise his colour judgment. Thus for a person whose job demands instant and accurate appreciation of coloured lights, e.g. traffic or navigation lights, most information is gained from a lantern test. For a colour blender or dyer, a matching test of some kind will probably be the most suitable. Yet in each of these examples there is much to be said for employing more than one kind of test before making a final diagnosis. The Admiralty use a procedure which incorporates a confusion test (Ishihara) and a lantern test. In the first test the candidate is light-adapted and is not asked to name any colours, whilst in the second he is dark-adapted and the colours must be named. Despite its value for recognising the existence of defective

colour vision, the typical lantern test, when used alone, employs too few colours for any precise diagnosis of the nature of the defect to be possible.

In the U.S.A., opinion is veering in favour of the application of a battery of tests involving varied testing procedures before a final assessment of any colour deficiency is made. It is a mistake to rely solely on the result of a single test, as is illustrated by the experience of a candidate for the Royal Air Force, which was recently described in a letter to the *Sunday Times*. He failed the Ishihara test and was rejected, but after vigorous protests was re-examined on other equipment, and eventually accepted and was able to take up a career as a pilot. Such an experience indicates the dangers that may arise if rapid testing of school-children's colour vision becomes part of school medical inspection. Failure to pass a confusion test may have profound effects on the career of a child by preventing his entry into an occupation in which the extent of his colour defect might not be sufficient to prevent him from holding down the job successfully.

It seems reasonable to suggest that a well-known pseudoisochromatic test, e.g. the Ishihara or the Stilling charts, can with advantage be followed by one which gives more information of the degree of colour weakness and, if possible, the standard of colour discrimination. The two Farnsworth tests are very useful here. The Farnsworth-Munsell 100-Hue test³ is extremely comprehensive and is one of the best tests available at present for judging colour discrimination. By following the elaborate system of graphical scoring of errors in the order in which the plastic chips are placed, much valuable information may be obtained of the degree of colour discrimination and of any defective colour vision. It is easy to ascertain in which parts of the chromatic circle the confusion zones of the examinee lie. One interesting possibility is to discover that a person having normal colour vision may make more error scores than one who is known to have defective colour vision (detected by other colour blindness tests), owing to inferior colour discrimination. The Farnsworth Dichotomous test⁴ is a much simpler version of the 100-Hue test, and there is only one panel of plastic chips to arrange in the correct order, instead of four. Yet it will differentiate between a slight colour weakness and a severe one, and thus is of obvious value in helping to decide whether a slight abnormality in a person's colour vision is sufficiently serious to prevent him from satisfactorily performing a routine job in certain industries.

Other tests have been developed which are more sensitive than the typical confusion test to slight differences in colour perception. These include

the Garner test³, the Glenn Colorule³ and the Wain Dichroic Ladder test². Wain has used his test in conjunction with the Ishihara to examine the colour vision of a large number of young people. Both the Wain test and the Glenn Colorule are able to detect slight variations from normal colour vision, but much more work must be carried out on these two devices before the results can be fully interpreted. In Garner's test, however, any confusion-matching zones are related to the matching positions of the test patterns in the colour triangle. Greater refinements in pseudoisochromatic tests will in turn lead to a fuller understanding of the results obtained from the Glenn Colorule and the Wain test. The ingenious use of a Wain floater and matching pattern by McLaren⁴ is another example of the use of a metameric match as a means of investigating defective colour vision. McLaren's suggestion is to match the two patterns by varying the colour temperature of the illuminant instead of fixing the illuminant and sliding the floater pattern alongside the range of patterns until a match is made.

Some pseudoisochromatic tests contain a large number of plates, since this used to be regarded as essential to reduce the possibility of memorising the correct answers. Thus the 8th edition (Japanese) and 9th edition (British) of the Ishihara test comprised 32 plates, and the 10th edition (Japan-1951) 38 plates. Surprisingly, the Ishihara test has been criticised because of careless publication of the key to the plates, which is an obvious help to anyone who wishes to learn by heart the correct answers. The 21st edition of the Stilling test (1952) has 37 plates and contains most elaborate instructions to avoid abuse by malingerers, i.e. those having normal colour vision who wish to be classified as colour defectives, and colour-blind people who desire to conceal their defect. In 1940 the first edition of the American Optical Company's Pseudoisochromatic Plates was published. This consisted of 46 plates and it also contained instructions for the detection of malingerers.

In recent years, the necessity for having such a large number of plates (many containing double letters or figures) in the Ishihara and Stilling tests has been queried by Pickford², Hardy, Rand and Rittler², and others. They point out that many plates are capable of alternative interpretations and that a smaller number of carefully selected plates would give more reliable results. The test developed by the American Optical Society, which appears to contain plates taken from the Ishihara and Stilling tests, contains several plates which have similar hues and similar confusion figures. When the test was revised in 1945, the number of plates was reduced to 18, and these were claimed to give results at least as reliable as those from the original 46. This revised test was in turn superseded in 1955 by an entirely new set of plates known as the A.O.H.-R.-R Pseudoisochromatic Plates for Detecting, Classifying, and Estimating the Degree of Defective Colour Vision⁵.

The test is based on the research of Hardy, Rand and Rittler, and relies on confusion with grey by

the various classes of colour-blind observers. Although the title implies rather sweeping claims for the diagnosis of defective colour vision, the test is simple to carry out. It is claimed that the test is particularly useful for testing school-leavers, and attention is drawn to the fact that mild colour defectives (as classified by the test) should not be excluded from certain occupations. Such persons are able to identify correctly the colours of electric wires, radio and television resistors, and so on. This supports a statement made earlier in this paper.

There are 24 plates altogether, of which the first four are demonstration plates to familiarise the subject with the test. Every plate has a background of grey dots which vary in size and depth (brightness); there are various shapes (circles, triangles, crosses) coloured in the hues that are confused by the usual types of colour defectives, viz. reds, greens, blues, yellows, etc. The results of the tests, except those from the four demonstration plates, are scored on carefully compiled record sheets. These show the error made in reading each plate by a protan—using the word in its modern collective sense to cover red-blind, protanopic, or protanomalous—deutan, tritan, and tetartan. The last is a possible type of blue-yellow deficiency whose existence has yet to be established. In any case, blue-yellow defects are very rare⁶.

The first four demonstration plates are easily read by all observers, but they give experience of the shape of the symbols to be encountered in later tests. They are followed by six screening plates, two of which sort out the blue-yellow defectives and the remaining four the red-green defectives. If the correct response is given in all cases, the candidate is deemed to have normal colour vision, and for him the test ends here. Any errors represent a qualitative diagnosis of a colour-vision defect in either the red-green or the blue-yellow region, and the test now proceeds with the reading of two alternative sets of plates, depending on the preliminary classification. Thus red-green defectives are shown plates No. 7-16 and blue-yellow defectives No. 17-20. The responses here give a quantitative grading as mild, medium, or strong protan, deutan, tritan, or tetartan, depending on the symbols seen. These gradings result from printing the figures in increasingly saturated hues. Mild defectives fail to see only the coloured figures of low saturation but perceive the others; severe (strong) cases cannot see even the colours of highest saturation.

Although, at first sight, there seem to be almost as many plates in the A.O.H.-R.-R test as in (say) the Ishihara, it will be appreciated that, whereas in the latter test all plates are shown to all observers before an assessment can be made, this is not so in the former test. Excluding the four demonstration plates, the value of which is obvious to anyone who has used the test, the number of plates required to be shown in the A.O.H.-R.-R test is—

Normal subjects	6
Red-green defectives	6 + 10 (diagnostic) = 16
Blue-yellow defectives	6 + 4 (diagnostic) = 10

These numbers cannot be regarded as excessive when it is considered that, at the end of the test, the candidate's colour vision has been checked and shown to be either normal or abnormal to a varying degree of red-green (protan or deutan) or blue-yellow (tritan or tetartan) blindness.

It is easier to criticise a colour-vision test than to devise one, and the A.O.H.-R.-R test has naturally been subjected to critical examination, particularly in America, and certain potential defects have been revealed. Nevertheless, the test is rated high among confusion tests and, on the whole, the ambitious claims made for it seem to be substantiated. One point not previously mentioned is that it is imperative to carry out the test under lighting that corresponds to Illuminant C, i.e. a colour temperature of approximately 6700°K. A good modern daylight colour-matching lamp is suitable.

Although it is not a great disadvantage to have to use the A.O.H.-R.-R test under special lighting conditions in order to obtain correct results, it would obviously be helpful to have a colour-vision test that could be operated under any customary form of lighting, including ordinary electric light. Such a test is the Illuminant-Stable Color Vision test⁷. Compared with the A.O.H.-R.-R test it is rather brief, but the plates have been designed to be usable under any conditions of normal illumination. Thus if the test is used under tungsten lighting, the results are valid, whereas other tests yield unreliable results.

The 2nd edition (1952) contains 6 plates, each of which has a number (or numbers) of a different hue from the background of coloured dots, the principle being similar to that of the Ishihara and other confusion tests. The plates have been specially designed to avoid a failing in some Ishihara and Stilling plates, namely, that a colour-blind person might be able to give the correct answers by judging the shape or form of the letters and not by colour perception. Blue blindness is ignored in this test, as being of no practical importance, but other types of colour defectives are readily separated from normal persons, since they see no figures, whereas normals read them all. The test therefore combines brevity with speed, as well as independence of the conditions of illumination. A later edition has a seventh plate containing three

designs, which is included for special diagnostic purposes.

Practical Examination of Colour Vision

Some time ago I was asked to report on the colour vision of some members of the staff of a firm of commission dyers. Four of these (A-D) were connected in one way or another with passing shades, and the other two (E, F) were foremen dyers. After some consideration the following tests were carried out, using the light from a Siemens colour-matching unit F.T.F. 2444. Each test was arranged so that the eye of the observer was at right angles to the plates, panels, or patterns, on which the illumination fell at an angle of 45°.

1 Confusion Test

The Stilling test was chosen because it seemed to embody several testing techniques, some of which might be of help in subsequent tests by giving a preliminary indication of any slight differences in colour vision. It contains several pseudoisochromatic plates, a few Vexier (puzzle) or pseudo-nonisochromatic plates, some plates (K1-K7) based on increasing colour contrast, and others (F1-F4) based on diminishing colour contrast.

2 Dichotomy Tests

These were used to separate those having normal colour vision from those having some form of defective colour vision—

- (a) The Farnsworth Dichotomous test D.15
- (b) The Illuminant-Stable Color Vision test (Freeman)
- (c) The A.O.H.-R.-R Pseudoisochromatic Plates.

3 Matching Tests

The matching tests were used to give a comparison of the degree of colour discrimination—

- (d) The Farnsworth-Munsell 100-Hue test
- (e) The Wain Dichroic Ladder test (using ranges No. 2, 3, and 4).

The above tests cover different aspects of colour judgment, so that it would have been surprising if the collective results had been completely consistent. In view of the occupation of the six observers, any serious deficiencies in colour vision were hardly to be expected, but it seems clear from

TABLE I
Summary of Results

Observer	Age Group	Stilling test	Farnsworth Dichotomous test	Freeman & A.O.H.-R.-R tests	Farnsworth-Munsell 100-Hue test (for colour discrimination)	Wain test (matching position on each range)
A	50-55	No defect	Correct	Normal	Good	5-6
B	55-60	Red-green defect	Correct	Mild red-green defect	Fairly good (Fig. 1)	10-11
C	45-50	No defect	Correct	Normal	Good	4-5
D	55-60	No defect	Correct	Normal	Good	5-6
E	50-55	No defect	Correct	Normal	Good-very good	5-6
F	30-35	No defect	Correct	Normal	Very good	4-5

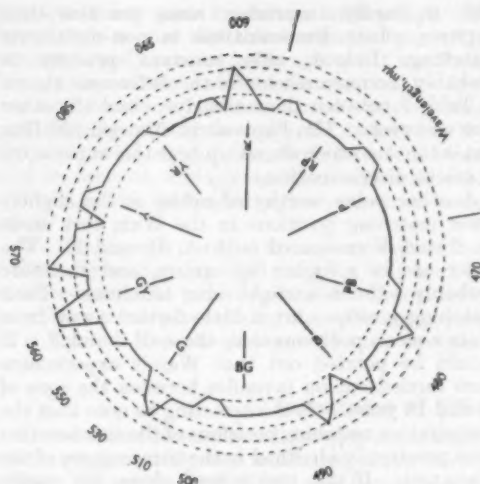


FIG. 1—Pattern from observer B

the tests that observer B is a mild colour defective, having moderately good colour discrimination. It is interesting to note that the mistakes made by B were not always those predicted by the compilers of the respective tests.

Stilling Test

B read two or three numbers incorrectly from the first batch of pseudoisochromatic plates, thus giving an indication of red-green colour-blindness. Yet he was not misled by the Vexier plates and gave correct answers to the K and F series, which the compilers regard as very sensitive. Plainly

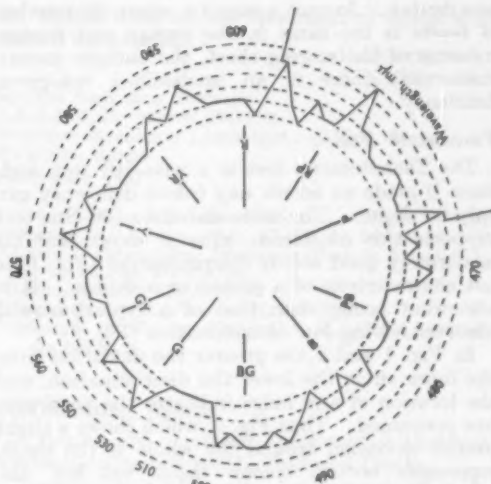


FIG. 2—Pattern of typical observer with normal colour vision but having low discrimination

his colour vision was only slightly abnormal according to this test.

Freeman Test

B read the figures in four of the six plates but not in the other two, whereas the compilers state that normals read all the plates and the colour-blind virtually none. This result also suggests that B's colour-vision defect is only slight.

A.O.H.-R-R Test

In the diagnostic test B failed in two tests, one of which classified him as a protan and the other

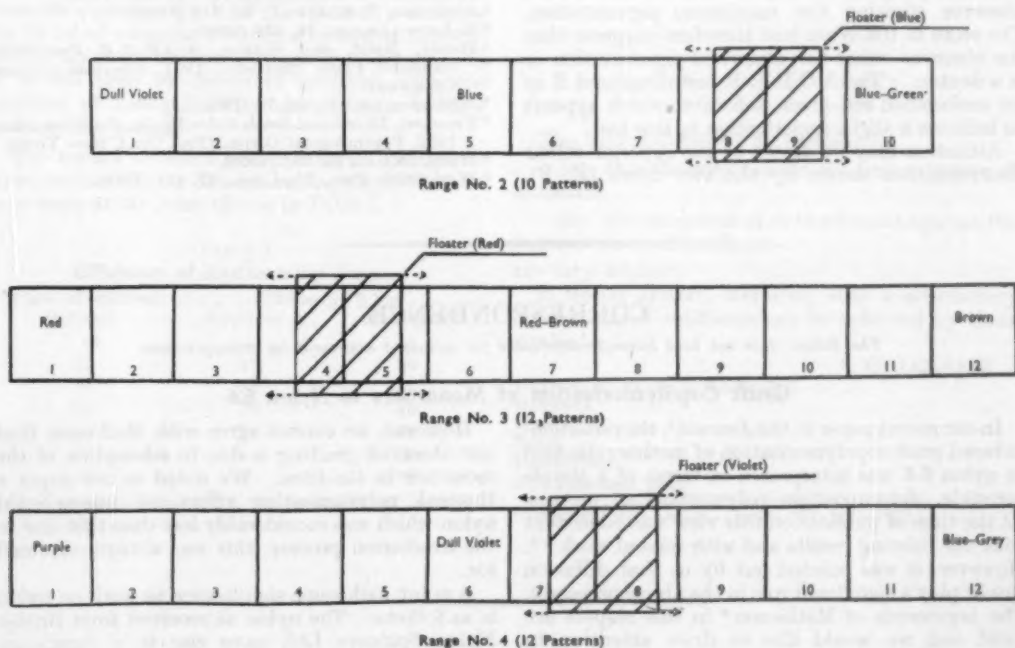


FIG. 3—Method of using the Wain test

as a deutan. In such a case, i.e. where the number of faults is the same in the protan and deutan columns of the scoring sheet, the authors merely state that there is an unclassified red-green deficiency.

Farnsworth Test

The Dichotomous test is a pass-fail test and, since B made no errors, any colour deficiency can only be slight. The more elaborate 100-Hue test supports this conclusion, since it shows that his moderately good colour discrimination (Fig. 1) is not characteristic of a protan or a deutan. It is somewhat better than that of a typical normal observer having low discrimination (Fig. 2).

In Fig. 1 and 2 the greater the deviation from the inner circle the lower the discrimination, and the location of any bulge indicates the particular hue concerned. Thus Fig. 2, which shows a slight overall deviation around the whole of the circle, represents normal colour vision but low discrimination. In Fig. 1 the pattern conforms rather more closely to the inner circle than in Fig. 2, thus suggesting a somewhat higher discrimination.

Wain Test

B is clearly out of step with the others in this matching test, and is much further away from the mean match as determined by Wain². His matching position (10-11) is the same in each range (blue, red, and grey) and four or five positions away from that of his colleagues. One would expect some differences in passing shades, compared with his colleagues.

Warburton³, using a similar test, found that a protanope gave a normal reading, whereas a deutanope gave one beyond that of the normal observer showing the maximum pigmentation. The score in the Wain test therefore suggests that the observer either has abnormal pigmentation or is a deutan. The A.O.H.-R-R test diagnosed B as an unclassified red-green defective, which appears to indicate a slight imperfection in this test.

Attention may be drawn to the superior colour discrimination shown by the two dyers (E, F).

This is hardly surprising, since practice does improve colour discrimination in non-metameric matching. Indeed, this constant practice is probably the main reason for the differences shown in Table I between these two dyers and the other four observers. The Farnsworth-Munsell 100-Hue test is the one which shows up best this superiority in colour discrimination.

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(Received 9th April 1961)

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However, we cannot agree with Mathieson that our observed grafting is due to adsorption of the monomer in the fibre. We noted in our paper a thermal polymerisation effect on unirradiated nylon which was considerably less than that due to the irradiation process; this was always corrected for.

A point with some significance to work on nylon is as follows. The nylon as received from British Nylon Spinners Ltd. gave rise to a significant thermal grafting with methacrylic acid in the

absence of any prior irradiation with γ -rays. We tentatively suggest that this was due to "peroxidic" centres already present in the nylon. It was found that nylon slowly oxidised ferrous ion to ferric ion and iodide to iodine. On destroying these peroxides by heating the nylon for 24 h at 100°C in the absence of oxygen, the above reactions ceased and the amount of blank reaction was considerably reduced. This preheated nylon was used in our work¹.

We would point out that the nylon received from British Nylon Spinners Ltd., and for which we are indebted, was the purest available. Peroxidation may have taken place during the spinning process. It is obvious that the presence of such peroxides

can vitiate work on the treatment of nylon with various monomers.

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Assessment of Silicone Antifoaming Agents

The occasion arose where I found it necessary to assess comparatively two antifoams of the silicone type. The article by Hadfield and Lemin¹ on the effect of foam on the rate of flow of liquor through a package suggested a method by which this might be done.

The apparatus used initially was a commercially available laboratory dyeing machine which possessed most of the features previously described, including a means of measuring the rate of flow. The package was constructed from 15 g of scoured cotton staple, compressed into the form of a cartridge 2.5 cm \times 9 cm by means of a hydraulic ram so that a uniform package was always obtained. Lissapol NX (1 g/l.) was used as a foam creator throughout and various concentrations of antifoam were used. Trials were conducted at different temperatures, pH and water hardness, and the efficiency (E) of the antifoam was calculated from the equation $E = 100P/T$, where P is the time for 50 ml of solution, without addition of Lissapol NX or antifoam, to flow through the package, and T is the time for 50 ml of solution, with the addition of Lissapol NX and antifoam, to flow through the package.

The results obtained under different conditions were similar. Those obtained using distilled water as a basis at 30°C are shown in Table I.

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Concn. of antifoam (p.p.m.)	Efficiency (%) of	
	Antifoam A	Antifoam B
0*	—	—
10	47	39
20	54	43
30	57	46
40	91	49
50	—	60
60	—	83

* In presence of Lissapol NX but in absence of antifoam $E = 28\%$.

The results are in strong agreement with those previously obtained by Hadfield and Lemin.

Another apparatus was constructed of non-conducting materials, and electrodes were mounted on either side of the package. To prevent earthing through the system, the liquor was forced through the package with compressed air instead of a pump. The basic solution used was 0.1-M potassium chloride plus 2 g/l. Lissapol NX. The resistance between the two electrodes was measured, while the liquor was flowing, for various concentrations of antifoam. Only one antifoam was examined and the results obtained are summarised in Table II.

TABLE II
Electrical Resistance of Solution containing Antifoaming Agent

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0	371
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20	286
30	244
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The curves obtained by plotting—

(a) the efficiency against the concentration of antifoam

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are very similar.

It would appear, therefore, that a satisfactory comparison of antifoams can be achieved by these methods.

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Notes

Society Honours

At its meeting on 8th November 1961 Council made the following awards, on the recommendation of the Medals Committee—

<i>Gold Medal</i>	C. L. Bird	for Exceptional Services to the Society and for Research in the Tinctorial Arts
<i>Silver Medal</i>	W. C. Castle	for Valuable Services to the Society in Australasia
	A. W. Doyle	for Valuable Services to the Society
	H. Hampson	for Valuable Services to the Society
	J. C. Wright	for Valuable Services to the Society

Council Meeting—6th September 1961

Among the matters discussed at the meeting of Council on 6th September were—

COMMITTEES—Mr. M. R. Fox was appointed a member of Publications Committee, and Dr. J. K. Skelly a member of the *Review of Textile Progress* Committee.

REPRESENTATION—Mr. J. P. Gill was confirmed as the Society's representative on B.S.I. Committee S/27—Record Inks.

DIPLOMAS REGULATIONS—On the recommendation of Diplomas Committee certain changes were made in the Diplomas Regulations. Details are given in a note below.

MEMBERSHIP—12 Ordinary Members and one Junior Member were elected.

Diplomas Regulations

At the meeting of Council held on 6th September it was resolved that the Diplomas Regulations be amended by the addition to Rule 3 of the following—

"alternatively a candidate who has taken a part-time course of instruction while receiving practical works training under the supervision of his employer may be allowed to take Papers D and E(i) or E(ii) at the conclusion of his course of instruction provided that he satisfies the Council, by submission of a full and detailed statement from his employer of the practical training he has received, that this training has been adequate preparation to enable him to attempt Papers D and E(i) or E(ii), but such a candidate after satisfying the examiners in all the necessary written papers may be required to serve to the satisfaction of the Council a further period of approved service in one or more branches of tinctorial technology before the Associateship can be awarded."

It was also resolved to insert in the Diplomas Regulations a statement to the effect that a candidate must take all the papers within four years and any candidate failing any one paper more than twice may be required to retake all the papers at any further attempt.

Associateship Examination 1962

Commencing with the 1962 A.S.D.C. Examination, Paper A will be divided into two separate papers. Paper A1 will be on "General Chemistry and Properties of Dyes, Pigments, and Auxiliary Products" and Paper A2 on "General Chemistry and Properties of Organic High Polymers". The Examination will therefore consist of six papers.

Papers A1, A2, and B will be held on Friday and Saturday, 25th and 26th May, 1962. Papers C, D, and E(i) or E(ii) will be held on Friday and Saturday, 1st and 2nd June, 1962. Application forms, which must be returned by 28th February, are available from the General Secretary.

Colour and the Journal

To assist in the identification of particular volumes of the *Journal* and, incidentally, to brighten its outward appearance, it has been decided to introduce coloured printing on the cover, starting in January 1962. The colour will be changed annually.

Worshipful Company of Dyers

At a General Court of the Company held on 11th October for the election of Wardens for the ensuing year, Mr. Derrik Leonard Bryant Marshall was elected Prime Warden and Mr. James Blair, B.Sc., F.R.I.C., was elected Renter Warden.

Diffusion and Mass Transport in Solids

The Institute of Physics and The Physical Society is holding a conference on this subject at Reading on 10th and 11th April 1962. It is provisionally proposed to hold sessions on general theoretical aspects of diffusion in solids and on diffusion and related mass transfer phenomena in metals, in oxides and covalent materials, and in strongly ionic solids. Details will be available in January 1962 from the Administration Assistant, The Institute of Physics and The Physical Society, 47 Belgrave Square, London S.W.1.

International Knitting Machinery and Accessories Exhibition, Manchester, October 1961

New Developments in Dyeing and Finishing Machinery

It was surprising to find so high a proportion of dyeing and finishing machinery in what was essentially an exhibition of knitting machinery. An outstanding feature was the increasing use of automatic control and process sequence programming on a wide variety of machines. It is, moreover, obvious that the maker of dyeing machinery is taking a responsible share in the development of new processing techniques.

Stainless steel is now the universal constructional material for dyeing machines, although a foretaste of possible new developments was given in the use of titanium for certain sections of a chlorite bleaching plant.

Package-dyeing machines were featured by all the leading manufacturers, the main emphasis being on machines capable of operating at temperatures of up to and above 130°C. In most of these machines the centrifugal circulating pump has been replaced by a reversible pump of the turbo type, which eliminates the reversing valve. The use of compressed air for the application of static pressure has given place to an auxiliary booster pump, which also provides a means of injecting dye into the system. Simplified locking of the pressure chamber by a single control is also a feature of the latest designs. Package-dyeing machines, both open and pressurised, have been widely adopted for dyeing knitted fabrics on beam, and such machines were a conspicuous feature of the exhibition.

Certain firms featured a hank carrier for use in their package machines, but the majority of the hank-dyeing machines were of the Hussong type, with a number of interesting developments. The introduction of bulked synthetic-fibre yarns has necessitated the provision of a second set of sticks to retain the bottom of the hanks in position. The distance between top and bottom sticks is adjustable, in some cases by a single control, and the sticks in each row are often staggered. In one machine the liquor circulates under pressure through the upper hank poles and thence into and through the hanks. In another design, circulation is improved by the provision of a weir parallel to each of the four sides of the machine in place of the traditional weir at one end only. This machine is also fitted with a self-sealing lid enabling a temperature of 218°F to be attained.

The Maxwell Colour Centenary

About 220 people from twelve countries attended the Maxwell Colour Centenary Conference, held at the Imperial College of Science and Technology, London, on 16th-18th May under the joint auspices of the Colour Group (Great Britain) and the Inter-Society Color Council (U.S.A.). The centenary being celebrated was that of the lecture delivered by Maxwell at the Royal Institution on 17th May 1861, when he gave the first public demonstration of three-colour reproduction by projecting images in red, green, and blue light in superposition on a screen. In the centenary discourse on "A Hundred Years of Colour Photography" held at the Royal Institution exactly 100 years after Maxwell's

The latest designs of totally enclosed side-paddle machines for hosiery and garments were to be seen on a number of stands. In addition to multi-speed drive, one such machine was fitted with extendable paddle blades. A new design in rotary dyeing machines was shown in which the drive gears had been removed to the outside, every part inside the tank being of stainless steel. Considerable interest has been shown recently in combined dyeing and boarding machines for ladies' hose, and two such machines were on display. In both cases the hose are placed on vertical flat metal forms on which they are carried through a series of steam setting, dyeing, and drying processes. The machines differ in the sequence of operations, and in the method of applying the dye solution, in one case by spraying and in the other by total immersion. All processing is automatically programmed and controlled.

A number of machines for continuous dyeing were on show, the main features of which were precision control of the padding mangles and very low pad-bath capacities. In certain cases the dye solution was carried on top of the pad rollers. Two installations incorporated a continuous pressurised steamer, entrance and exit being sealed by nip rollers. A continuous bleaching range for knitted fabrics and a continuous process for the shrink-resist treatment of wool tops were also featured.

One of the most important recent developments in the dyeing of textiles has been the introduction of perforated drum dryers, and several examples of this type of equipment were on exhibition. This form of dryer is particularly suitable for drying individual garments, hanks of yarn or loose stock, which are held in place on the drum surface by the suction of hot air in its passage from the outside to the interior of the drum.

A wide variety of equipment for calendering and stentering knitted fabrics was on display, the chief features being higher speeds and automatic control at every stage. An important trend in recent years has been the development of brushed and raised knitted fabrics, and a number of machines suitable for raising fabrics of both synthetic and natural fibres were exhibited.

The 1961 Exhibition was undoubtedly a landmark in the development of the Midlands textile industry.

H. W. PARTRIDGE

lecture, Dr. D. A. Spencer explained how, by fortuitous circumstances, Maxwell was able to produce a three-colour photograph some 20 years before photographic emulsions sensitive to the green and red had been produced.

The theme of the Conference progressed from a study of the facts of three-colour mixture and their biological interpretation to their application in photography, television, and printing, with a final day devoted to a discussion of colour appearance.

A summary of the Conference, by Professor W. D. Wright, appeared in *Nature*, 191, 10-11 (1 July 1961).

West Riding Section Junior Members' Competition

The Dyers' and Finishers' Association have donated the sum of 30 gns. to the Society for the 1961-62 competition for Junior Members resident in the West Riding. This will be awarded as prizes to the value of 15 gns., 10 gns., and 5 gns. for the three best essays submitted.

Essays are invited on any one of the following subjects—(1) the influence of preparation on subsequent dyeing and finishing processes, (2) the synthesis of polymers within textile fibres and the effects obtained, (3) the advantages and disadvantages of dyeing at temperatures above 100°C, (4) new methods developed for dyeing and finishing materials composed of acrylic fibres, (5) the extent to which the dyeing and finishing of man-made fibres has borrowed from the techniques used for wool, and *vice versa*.

Details of the competition rules and entry forms can be obtained from the Honorary Secretary of the West Riding Section, Miss Esmée Smith,

Flat 16, Foxhill, Weetwood Lane, Leeds 16. The closing date for entries is 1st February 1962.

Chemical Society Library

The Library will close for the Christmas vacation at 7.30 p.m. on Friday, 22nd December, and will reopen at 9.30 a.m. on Thursday, 28th December.

Meetings of Council and Committees October

Terms and Definitions—2nd

Conference Subcommittee of International Relations—6th

The Worshipful Company of Dyers Research Medal—13th

Publications—17th

Fastness Tests Co-ordinating Committee—Carpet Subcommittee—24th

Finance and General Purposes—24th

Examinations Board—26th.

New Books and Publications

Industrial Organic Nitrogen Compounds

By M. J. Astle. Pp. vii + 392. New York: Reinhold Publishing Corpn. 1961. Price, 112s. 0d.

The subject matter of an American Chemical Society monograph should, according to that society, include a complete and critical treatment of a relatively restricted area of knowledge. In the present case a brave, and mainly successful, attempt is made to cover the extensive field of modern organic nitrogen chemistry. However, such an attempt is almost bound to lead to a product in which deficiencies may be found. This comprehensive approach by the author results in a book which consists, to a large extent, of one-sentence descriptions of chemical reactions. This may be admirable for a work of reference, but one is left with the feeling that a little more detail would have added far more than the space it would have occupied. To quote one example at random (p. 136)—it is stated that γ -pyrone is converted into γ -pyridone on treatment with ammonia at high temperatures. No indication of the mechanism of this process is given, although the author states that reaction mechanisms are discussed whenever they appear useful in understanding the chemistry involved.

The book is well-produced with an excellent clarity of type and formulae, and contains very few printer's errors. The first three chapters are devoted to aliphatic, aromatic, and heterocyclic amines and, together with a chapter on hydrazines, azo compounds, diazonium salts and oximes, make up two-thirds of the book; this is a balanced proportion in a work concerning industrial nitrogen

compounds. The section on heterocyclic amines gives a concise account of 3-, 4-, 5- and 6-membered saturated and unsaturated ring systems containing one or two nitrogen atoms and the derivatives of such systems. Thus the chemistry of the pyrazoles, imidazoles, pyrazines, and pyrimidines is fully described and the chapter can be regarded as a good introduction to heterocyclic chemistry in general. The remaining four, shorter, chapters are given over to nitriles, amides and amino acids; isocyanates and ureas; aromatic nitro and nitroso derivatives; and aliphatic nitro compounds. Each chapter is well illustrated with formulae and the references (1688) include a number taken from patent literature.

A fundamental criticism which can be levelled is that the diazohydroxide, rather than the diazonium salt, is stated to be the active agent in a coupling reaction (p. 206). The only evidence referred to, for this statement, is dated 1895, and no account of modern theories concerning the nature of the coupling entity is given. References to *s*-triazine derivatives are very brief, and no mention is made of cyanuric chloride and its present-day importance.

In spite of these shortcomings and in view of his very wide terms of reference, the author has more than achieved his aim in covering the field of organic nitrogen chemistry. The monograph can be recommended as an excellent source guide both to the research student and to those concerned with dye intermediates, especially since several years have elapsed during which no volume devoted solely to this subject has been published.

F. JONES

Mark Thornhill Wade, Silk Dyer, Soho

By D. B. Dagley. Pp. 157. London: The Research Publishing Company. 1961. Price, 16s. 0d.

Silk dyeing in Soho sounds an attractive subject, and Mr. Dagley has written an unusual and interesting book about his long-dead relative M. T. Wade, who was in business as a silk dyer from 1788-1802, thereafter leaving the trade owing to gambling losses in the State lottery.

The book reconstructs the history of an ordinary London family from contemporary records and M. T. Wade's business ledger. A description of the working life of the dyer is not attempted, although it is well paraphrased in a quotation from the Book of English Trades—"The business of a Dyer is laborious and chilly; the workmen are constantly dabbling in water hot and cold". There is a brief description of dyeing in 1788, but the factual part of the book is based on the ledger showing customers' names, materials, prices, and colours dyed. These items, classified and described, are of particular interest to garment dyers.

The main theme of the book lies in the side allusions which have nothing to do with dyeing: the case histories of customers, e.g. Mr. Angelo, who taught Lord Byron fencing; the neighbours, such as Mr. Sheraton and Mr. William Blake; the maps and sheet plans of the period; and what was happening in London on the day M. T. Wade dyed any particular order. And so it is a picture of M. T. Wade's times rather than his life as a dyer (his gambling life is well documented). Unfortunately the author has dragged in too many details and tit-bits, regardless of their significance to the story. There is too much surmise in the history, despite the research spent upon it. There are too many facetious comments by the author.

Mr. Dagley has shown that technical developments are only one side of the varied history of dyeing. The dyer of today, no less than M. T. Wade of 1800, cannot remain just a dyer—he is moulded by the stream of life around him.

R. A. PEEL

Scientific Thinking and Scientific Writing

By M. S. Peterson. Pp. vii + 215. New York: Reinhold Publishing Corp. 1961. Price, 56s. 0d.

Better English for Technical Authors

By T. W. Kirkpatrick and M. H. Breese. Pp. x + 122. London: Leonard Hill [Books] Ltd. 1961. Price, 14s. 0d.

These two books, the first by an expert in food technology and the second by two agriculturists, are at first sight as dissimilar as the proverbial cheese and chalk. They have, however, as a unifying theme the encouragement of better writing and it therefore seems appropriate to review them together.

Dr. Peterson, in his preface, boldly claims that good organisation is the key to good writing and that "bad grammar, misspellings, and incorrect punctuation may tarnish but cannot destroy the value of a well-organised scientific article, treatise, talk, or progress report." Mr. Kirkpatrick and

Mr. Breese, on the other hand, have been struck by the "bad style and grammar of many . . . theses and reports, often admirable in other respects", and have therefore compiled a list of the mistakes most commonly made and have written short notes on how they should be avoided. To a large extent, then, the two books are complementary.

Scientific Thinking and Scientific Writing is based on the postulate that good expository prose cannot be written without the application of the general logic of science. Chapter 1 discusses the logical flow of a scientific investigation; a clear and complete statement of the problem is followed by a hypothesis based on *deductive* reasoning as the solution; analysis, utilising *inductive* reasoning, of the experimental data leads to a final generalisation. The same procedure should be followed in writing a journal article. The next chapter deals with classic methods of inductive reasoning and their application to the organisation of scientific articles and reports; these methods are illustrated by means of examples. The author emphasises that inductive methods are used instinctively by the experienced investigator—he may not even know that specific methods of induction exist.

Chapter 3 describes some specific methods and techniques (biochemical, physical, microbiological, analytical, mathematical, and general methods used in industry) and their relation to experimental design and the interpretation of data, the purpose being to show the stepwise structure of such methods and to encourage the reader to examine them and thus to think creatively and logically about his own problems.

The final two chapters are more specifically concerned with scientific writing. In Chapter 4 "Building a Professional Background for Scientific Writing", great emphasis is placed on the value of obtaining a thorough knowledge of the classics of scientific writing (some 20 books are listed and their contents briefly described) and of cultivating good habits of observation. Chapter 5 discusses different types of scientific writing (essay, scientific talk, book review, literature review) and each type is illustrated by means of a model paper. In fact, over half the book is devoted to examples of good writing—it seems unnecessary, though, to have included the 201 references (6½ pages of text) that accompany the model literature review, since these are irrelevant to the particular use of the model.

Dr. Peterson emphasises that his book is not intended to be a profound treatment of logic or of scientific method. Occasionally simplification leads to an inference that is too wide. On p. 41, for example, it is stated that "if a piece of iron exposed to moist air rusts, we can infer that moist air is a cause of rust". This is not true, unless it has previously been ascertained that (a) moisture alone and (b) air alone does not cause rust to form. The book as a whole is, however, a very valuable contribution, and anyone who studies it carefully is unlikely to infer, from the statements "of the 20 (amino acids) tested . . . 9 were essential" (p. 14) and "the best results with protein are attained when a complete protein (one containing the so-called eight essential amino acids) is fed at each

meal" (p. 45); that "the older you become the fewer amino acids you need".

In *Better English for Technical Authors* the three main causes of poor writing are said to be the use of the wrong word, bad style (including inaccurate punctuation), and faulty grammar. Not all mistakes are equally bad; those that lead to ambiguity or force the reader to think twice before he arrives at the intended meaning are worse than "polysyllabic pomposities and circumlocutions", which in turn are worse than errors of grammar that offend the ear, even if they do not obscure the sense.

Part I is "mainly on style", Part II "mainly on grammar", and Part III deals with the elements

of punctuation and sundry subjects. The book is not intended to be a complete treatise on style and grammar; it concentrates on the commoner and grosser errors that the authors have observed in articles on agriculture and kindred subjects. Every book on style contains at least a few stylistic errors, and it is often tempting to the reviewer to search for these and in so doing neglect the value of the book as a whole. In this particular book not many have been found; most are trivial and do not merit specific mention here. In addition to its wealth of examples the book contains a very useful index and at its extremely reasonable price is well worthy of a place in the library of any prospective technical author. M.T.

New Books Received

- Moisture Determination by the Karl Fischer Reagent.* Second Edition. Pp. 16. British Drug Houses. 1961.
- Autographic Measurement of the Stress-Strain Properties of Dumb-bell Test Pieces.* A. G. BUSWELL. Pp. 11. Reprinted from *Trans. I.R.I.*, 37, pp. 20-29. Feb. 1961.
- The Xenon Lamp. An Evaluation of the Tendency of Rubber Compounds to Discolour on Exposure to Light and of PVC Colours to Fade.* A. G. BUSWELL. Pp. 11. Reprinted from *Trans. I.R.I.*, 37, pp. 43-51. April 1961.
- Natural Polymer Man-made Fibres.* C. Z. CARROLL-PORCZYNSKI. Pp. xi + 303. London: National Trade Press. 1961. 55s. 0d.
- Mark Thornhill Wade, Silk Dyer, Soho.* D. B. DAGLEY. Pp. 157. London: The Research Publishing Co. 1961. 16s. 0d.
- Investigation of Rates and Mechanisms of Reactions.* (Second Edition). Ed. by S. L. FRIESS, E. S. LEWIS, and A. WEISSBERGER. Pp. xii + 713. New York: Interscience Publishers Inc. 1961. \$23.50.
- Textile Series Report No. 114. Development of Shade Standards and Tolerances for Cloth, Wool, Cotton-filled Gabardine, USAF Shades Sage Green 522, OD 7A, and Blue 324.* Natick, Mass.: Quartermaster Research and Engineering Center, Textile, Clothing, and Footwear Division. Pp. vi + 57. 1961.
- Polypropylene.* T. O. J. KRESSER. Pp. xi + 208. New York and London: Reinhold Publishing Corp. and Chapman & Hall Ltd. 1960. 52s. 0d.
- Surface Activity.* J. L. MOILLIET, B. COLLIE, and W. BLACK. (Second Edition). Pp. xvi + 518. London: E. & F. N. Spon Ltd. 1961. 75s. 0d.
- The Technology of Washing.* G. R. PERDUE. Pp. vii + 251. Hendon: The British Launderers' Research Assoc. 1961. 42s. 0d.
- A History of 250 Years of Clothworking in London.* PERROTTS LTD. Pp. 48. Leeds, London, Huddersfield: Perrotts Ltd. 1960.
- The Fixation of Dyes containing a Dichloro-s-Triazinyl Group on Cellulose.* C. PRESTON and A. S. FERN. Pp. 9. Reprinted from *Chimia*, 15 (1961) 177-186.
- Reactive Dyestuffs for Cellulose.* I. D. RATTEE. Pp. 8. Reprinted from *Endeavour*, 20 (July 1961) 154-161.
- Five Language Dictionary (Paint, Varnish, and Lacquer).* R. SANTHOLZER and JAN KOŘIŠKÝ. Pp. 436. Prague: Orbis. 1959. Kčs. 20.20.
- Textile Recorder Annual and Machinery Review 1961-62.* Pp. 186. Manchester: Textile Recorder. 1961.
- XI Technische Landeskonferenz für die Textilindustrie. Kurzreferate.* Pp. vi + 137. Budapest: Textiltechnischer und Wissenschaftlicher Verein Ungarns. 1960.
- The Estimation of TiO₂ in Paper.* F. R. WILLIAMS. Pp. 12. Originally printed in *Paper-maker & Brit. Trade J.*, May 1950. 1961.
- Farbsysteme.* G. WYSZECKI. Pp. 144. Göttingen: Muster Schmidt Verlag. 1960. DM.21.
- Diazo and Azo Chemistry: Aliphatic and Aromatic Compounds.* H. ZOLLINGER. Pp. 444. New York: Interscience Publishers Inc. 1961. 124s. 0d.

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility for the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University.

Ciba Ltd.

CIBA REVIEWS. No. 141—This issue is devoted to mixtures of fibres. In addition to three articles dealing with the development of mixture cloths from medieval times to the nineteenth century, there is one on synthetic-polymer fibres in blended-fibre fabrics. The technical section includes notes on the use of benzyl alcohol in dyeing continuous-filament polyamide materials to produce dyeings free from barness and also as an addition to vigoureux printing pastes whereby steaming times can be shortened considerably.

No. 1961/1—This issue contains articles on the British calico-printing industry 1676-1840, the English contribution to the chemistry of calico printing before Perkin, the development of design in English printed textiles, and the influence of William Morris. These were originally prepared by the late Peter Floud and were revised and expanded by Mrs. Barbara Morris of the Victoria and Albert

Museum. Technical articles include one on the Cibaphasol technique.

No. 1961/2—This issue is concerned with colour and its measurement. There is a long article on this subject and two shorter ones on instrumental methods of colour measurement and the application of colour theory and measurement to the textile and dyeing industries. All are by Prof. W. D. Wright of Imperial College. The excellence of the articles, together with the abundance of diagrams, in colour as well as in black and white, makes this a most attractive and useful issue.

No. 1961/3—The theme of this issue is the use of metal threads for ornamenting textiles. One article describes the use of gold and silver for this purpose from medieval to modern times, while two others are concerned with the production of metallic threads by lamination and the production, properties, and uses of metallised textiles. A final article describes the production of bronze prints.

The technical section contains an article on testing light fastness in fading lamps. This refers to three new Fade-Ometers and three new Weather-Ometers incorporating 6000-watt water-cooled xenon arcs and a Sunshine Carbon Arc Lamp with a carbon arc source having a much lower U.V. emission and much higher emission in the visible.

Farbwerke Hoechst A.G.

NAPHTOL AS ON COTTON PIECE GOODS—This card contains dyeings on mercerised cotton cloth of the most important azoic combinations for use on this type of material. With the exception of the full-depth dyeings based on Naphthol AS-S, which in high concentrations is not recommended for padding, they have all been dyed continuously. Dyeings of no less than 475 combinations are included. In 125 cases the dyeings may be marked with the Indanthren label provided that they satisfy the minimum depth requirement.

REMAZOL DYE STUFFS ON MERCERISED COTTON YARN—This card contains dyeings of the Remazol reactive dyes on mercerised cotton yarn. The preferred alkali is a mixture of 4.5% of caustic soda (72° Tw) and 5% of soda ash (calc. on wt. of goods), dyeing being carried out at 50–55°C, except when using Remazol Turquoise Blue G, which gives maximum colour yield at 60°C. Ten dyeings of two- and three-colour mixtures are also included.

REMAZOL DYES ON NYLON-HELANCA KNITWEAR—This card contains dyeings on knitted cloth of twelve Remazol reactive dyes. These have been applied as follows. Dyeing is begun at 40°C in presence of 2 g/l. tribasic sodium phosphate crystals and continued at this temp. for 10 min. The pH is then adjusted to 6.5 by adding 2 ml/l. of acetic acid (30%). Depending upon the depth of colour, a further 1–4% of acetic acid (30%) is added, the temp. raised slowly to boiling point, and dyeing continued at the boil for 45–60 min. Deep dyeings may require the addition of a little formic acid to promote exhaustion. Remazol Black R requires the use of sulphuric acid for neutralising and dyeing. It is recommended that these dyes are used alone or in two-colour combinations. "Blocking" may occur with deep dyeings, and completely uniform dyeings may not be obtainable on some types of polyamide material.

INDANTHREN PRINTING BLACK HC—This vat dye is particularly suitable for printing by the Colloresin process. It is recommended for washing styles and particularly for curtains and awnings. Black prints on all goods may be marked with the "Indanthren" label. The greys produced on viscose rayon are somewhat redder than those on cotton. Fastness figures (as a black) on cotton include—light 7–8, washing (95°C) 4–5, chlorine 4–5.

THE CHROME DYES: SALICINE CHROME AND METACHROME DYES—This card contains dyeings in two depths of 23 Salicine Chrome dyes which are intended for application by the afterchrome process and 12 Metachrome dyes. The majority of both groups have been marketed previously under different names, a list of which is appended. Dyeings of eight acid dyes suitable for shading are also included.

The Geigy Co. Ltd.

FAST SHADES ON BAN-LON FABRIC—This card contains dyeings on Ban-Lon knitted cloth of 14 acid dyes selected because they adequately cover irregularities in the dyeing behaviour of the material, do not block-off, and give dyeings of good fastness to light. In certain cases, coverage of irregularities can also be improved by adding Irgasol NJ to the dyebath. The importance of correct preparation of the cloth before dyeing if irregularities are to be covered as completely as possible is stressed, and a scour for 15–20 min at 60–70°C with non-ionic detergent is strongly advocated.

Imperial Chemical Industries Ltd.

TECHNICAL INFORMATION LEAFLETS—

Dyehouse No. 600. Subject Index to Technical Information Leaflets (Dyehouse) Nos. 161–599.

Dyehouse No. 602. Effect of Metal-sequestering Agents in the Dyebath—As far as Procion dyes are concerned, only the highly active products, e.g. Irgalon BT, Trilon B etc. are liable to cause trouble. Water-softening agents of the

polyphosphate type, e.g. Calgon, have no adverse effect on the colour of the dyeing.

Dyehouse No. 617. Procionyl Dyestuffs on Nylon—Recipes for producing a very wide range of British Colour Council colours are tabulated.

Dyehouse No. 619. Pigments for Distempers and Emulsion Paints—Dry distempers may be coloured with both concentrated and reduced brands of pigments. Oil-bound distempers are very conveniently coloured by using the highly dispersed "V"-brand pastes. Organic pigments in alkyd emulsion paints may migrate from the aqueous to the oil phase with consequent loss of strength in the can and strength development on brushing. The technique for preparing stainers of oxides of iron, which are free from this defect, is described. Polyvinyl acetate emulsion paints may be coagulated by the "V"-brand pigment pastes. Methods for determining whether this is likely and, if so, of using Lissapol N as a stabiliser are described. Light fastness data on pigmented emulsion paints and recipes for particular colours are given.

Dyehouse No. 622. Viscose Rayon, Dyeing and Resin Finishing: Modified Continuous Piece-dyeing Procedures for Procion Dyestuffs—Viscose rayon cloth may be dyed continuously to all depths, free from surface frosting, using cold-dyeing Procion dyes and selected Procion H dyes. High-quality dyeings are obtained with the former, using the pad(bicarbonate)-batch-dry-wash sequence. When the cloth is to be resin-finished, the intermediate wash may be omitted and the dyeings obtained are of exceptionally high colour value, fast to wet treatments, and resistant to creasing.

Dyehouse No. 623. Dyestuff Division Products used in the Laundry Industry—Detergents, water-repellent agents, softening agents, fluorescent brightening agents, a dye-fixing agent, and cationic bactericides are listed.

Dyehouse No. 624. Fluolite MP in the Laundry—A liquid brand of this fluorescent brightening agent is offered. This is much more convenient to use than the powder brand.

Dyehouse No. 625. Procion Dyes on Cotton: New Processes for Dyeing and Resin Finishing—Exceptionally high colour value may be realised and dyeings of very good fastness and exhibiting excellent crease-resist properties obtained when cold-dyeing Procion dyes and selected Procion H dyes are applied continuously by the pad (bicarbonate)-dry-bake-resin finish-wash sequence. The unreacted dye is not removed before padding with the resin precondensate, since it is effectively fixed within the fibre by association with the resin. Addition of urea to improve colour yield is neither necessary nor desirable. Any of the normal resin precondensates may be used with only minor alterations in the composition of the padding liquor and no change in curing conditions.

Dyehouse No. 626 (Replaces No. 571). Use of Velan PF and Velan NW in the Production of Durable Oil- and Water-repellent Finishes on Textiles—T.I. 571 now appears to be out of date and the recommendations made in it have therefore been withdrawn. All queries on durable oil- and water-repellent finishes based on fluorochemicals made by the Minnesota Mining and Manufacturing Company should be referred to them.

Dyehouse No. 630. Cold-dyeing Procion Dyes on Viscose Rayon: Colour Yield by Various Dyeing Methods—The relative colour yields of 15 dyes applied by eight methods are tabulated.

Dyehouse No. 632. Emulsion Thickenings in Textile Printing: Possibility of Fire and Explosion Hazards during Baking (Dry-heat) Treatments—Potentially explosive air-solvent vapour mixtures can be formed when handling the solvents themselves and from the solvent-based emulsions. In the latter case, the operation most likely to give rise to potentially explosive conditions is the baking process. Although there is very little risk if normal precautions are taken, attention is drawn to the advice and facilities offered by H. M. Inspectorate of Factories on the minimising of hazards of this type.

Dyehouse No. 633. Autumn 1961 Fashion Colours—Nylon Hose (The Midland Hosiery Dyers' and Finishers' Federation).

Dyehouse No. 634 (Replaces No. 633). Light Fastness of Dyestuffs: ISO and AATCC Ratings—No official comparison system can be devised since the blue wool standards

are quite different. A conversion chart is reproduced which is generally reliable. The most reliable estimate of the AATCC sunlight rating is obtained from the ISO result obtained in Bombay. The ISO Wilmslow result (if different) should be used to provide the AATCC daylight rating. Because of the marked differences in the spectral characteristics of daylight and the light from a carbon arc, prediction of Standard Fading Hours from ISO results may be much less reliable.

CALEDON AND DURINDONE DYESTUFFS ON VISCOSERAYON—This card contains dyeings in continuous-filament viscose satin of 77 Caledon vat dyes, together with ten Durindone vat dyes dyed on viscose rayon yarn. Of the former, 66 can be used for Felisol-labelled goods; four of the Durindone dyes are also suitable. The usual comprehensive information on dyeing behaviour and fastness characteristics is appended alongside each pattern.

CALEDON AND DURINDONE DYESTUFFS PRINTED ON COTTON AND VISCOSERAYON—This card contains prints in two depths on cotton cloth and viscose rayon cloth of 60 Caledon vat dyes, 19 Durindone dyes, and Indigo 2R. The accompanying text (41 pp.) is particularly detailed and includes a tabular survey of methods of application,

usage, brands available, and notes on behaviour in printing. Eight examples of multi-coloured prints based on vat dyes and combinations of these with reactive, azoic, oxidation black and Alcan dyes are included.

FAST SHADES ON WOOL YARN—This card contains 54 dyeings on wool yarn. These have been produced using, singly and in combination, members of a range of seven Carbolan and 18 Coomassie dyes, Solway Violet BR, 5 Solochrome dyes, and Solway Blue Black B (dyed after-chrome). The recommended dyeing methods for the fast acid dyes are based on the use of Dispersol CWL as levelling agent with Silcolapse M437 added as an anti-foam. In the preferred method for the chrome dyes, all the acid is added to the water in the machine which is then circulated at 40°C for 10–15 min to give a pH of 4.5, the dissolved dye added, and dyeing continued. The dyebath is cooled to 80°C before the dichromate is added. The test includes a table of data on the exhaustion and levelling characteristics of each of the recommended acid dyes when dyed at pH 7 and pH 5.5 in presence of 3% of Dispersol CWL. The recommended temperature at which dyeing should be commenced at pH 7 is 100°C in many cases.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Continuous Automatic Control of Carbon Black Furnaces

Phillips Petroleum Co. USP 2,953,436 (14 Apr 1955)

The composition of the effluent gas is a definite function of the ratio of air to oil supplied to the furnace. In particular, the content of methane, acetylene, and hydrogen in the effluent decreases as the air:oil ratio increases. Apparatus is described by which the air:oil ratio is controlled automatically in response to automatic analysis of the effluent gas. C.O.C.

Carbon Black (C.I. Pigment Black 6 and 7) Pre-combustion-type Furnace having Improved Tangential Fuel Introduction

Phillips Petroleum Co. USP 2,961,300 (8 May 1958)

Pelleting of Carbon Black (C.I. Pigment Black 6 and 7)

Phillips Petroleum Co. USP 2,962,763 (5 Aug 1957)

Apparatus is described for spraying an aqueous solution of molasses on to the pigment at a constant rate. C.O.C.

Removing Grit from Carbon Black (C.I. Pigment Black 6 and 7)

Phillips Petroleum Co. USP 2,963,152 (28 Oct 1957)

Method and apparatus for removing the grit with less loss in pigment than has hitherto been possible. C.O.C.

Apparatus for Washing Loose Fibres

National Research Development Corp. BP 875,401 (2 Jan 1959)

The apparatus comprises an inclined chute having a water inlet at the top. There is an outlet for the water some way down the chute and means for carrying the fibres up the chute and also for creating turbulence in the water in at least one position along the chute. It is especially suitable for removing dirt and rotted vegetable matter from vegetable fibres obtained, e.g., by retting. C.O.C.

Wet Processing of Textiles, Paper, and the Like

Cocker Machine & Foundry Co. USP 2,964,825 (16 July 1958)

One of a pair of squeeze rollers has its axis above the level of the treating liquor. The axis of the other roller may be moved through an arcuate path of at least 180° with respect to the other squeeze roller. This enables the nip between the two rollers to have its position widely varied, e.g. so that material being treated may be either slop-padded or "drain" padded. C.O.C.

Continuous Steaming of Continuous Textile Fleeces

Fleissner & Co. BP 873,992 (Germany 21 Mar 1958)

The continuous fleece, which may be composed of strips, small pieces of fabric, or fibres, is passed through a steam-filled chamber while escape of steam from the entry and exit is prevented. During its passage through the chamber the fleece passes partly around at least one rotary drum sieve, the steam being sucked into the interior of the drum as the fleece passes over it. C.O.C.

Coating and Impregnating Fabric, Paper, or Plastic Sheet

J. H. Shelton USP 2,960,963 (15 Apr 1958)

The tanks containing the treating liquor or liquors are arranged in tandem so that whenever the liquid in one tank has to be modified or removed, the cloth or other material being treated may be lifted out of that tank and placed in the adjacent tank containing the same solution. This enables changes in the liquor in a tank or the cleansing of the tank to be carried out without interfering with the continuous movement of the materials under treatment through the processing line. C.O.C.

Devices for Loading and Unloading Rollers in Calenders, etc.

VEB Erste Maschinenfabrik Karl Marx Stadt BP 873,902 (31 Jan 1958)

Mounting Wire Teasles for Raising Machines

Lumb, Walshaw & White BP 873,991 (26 Nov 1958)

Wire teasles are so mounted on the drum that while they cannot freely rotate about their own axes, they do yield to a limited extent around their axes under the dragging influence of the cloth being teased. C.O.C.

Preventing Bonding of Doctor Rollers in Screen-printing Machines

Zimmer's Successors Co. USP 2,965,020 (24 June 1955)

Brush-coating Machine having one or more Oscillating Brushes

Agfa BP 875,779 (Germany 28 June 1958)

The machine contains devices to prevent or damp vibration and disturbance set up by increasing the velocity of the brushes and which can cause reduction in the quality of the finished coating. C.O.C.

Wet Processing of Textiles in a Fluidised Bed (VIII p. 583) Applying a Seamless Coating of Photosensitive Material to Printing Cylinders (IX p. 584)

Boarding of Knitted Articles in a Fluidised Bed (X p. 587) Calendering Unit for Production of Uniform Graining in Variegated Sheets (XIII p. 591)

II—WATER AND EFFLUENTS

Water Supply for the Textile Industry

K. Wuhmann *Textil-Rund.*, 16 (July 1961) 355-364
A survey, covering sources, means, and economics of water supply. P.B.S.

Waste Water Economy

M. Kehren *Textil-Rund.*, 16 (July 1961) 372-390
A survey of waste-water purification methods for the textile industry: methods of clarification, the effect of detergents on this, and the construction of clarification installations. The survey concludes with extracts from the Swiss and West German laws on disposal of waste water. P.B.S.

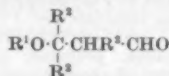
Adsorption Purification of Effluents containing Aromatic Compounds from Aniline Dye Plants

R. M. Becker, A. M. Koganovskii, N. N. Krayukhina, N. P. Myshkina, P. N. Taran, I. A. Troyanov, and S. M. Sheyn *Ukrain. khim. zhur.*, 27 (No. 2) (1961) 268-273
The contamination of effluents of aniline dye plants with nitro- and chloronitro-aromatic compounds necessitates the application of special purification methods. The use of activated carbon provides an efficient method. The regeneration of carbon requires steam treatment for the removal of volatile aromatic compounds and rinsing with 5% alkali for the removal of phenolic- and non-volatile compounds. I.G.

PATENTS

Preventing Growth of Slime-forming Micro-organism in Industrial Water

Shell International Research
BP 873,951 (U.S.A. 20 Apr 1959)
Addition of a compound of formula



(R^1 = hydrocarbon; R^2 = H or hydrocarbon), e.g. β -allyloxypropionaldehyde, effectively prevents formation of gelatinous substances, e.g. paper slime, in industrial waters.

BP 874,064 (U.S.A. 20 Apr 1959)

Esters of formula



(X = Hal; R = subst. or unsubst. hydrocarbon), e.g. methyl or ethyl α -chloroacetate, are used. C.O.C.

Selection of Fabrics for Filtration Processes (VI p. 577)
Polyvinyl Glycolic Acid Ether (Carboxymethylation of Polyvinyl Alcohol) (XIII p. 591)

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Table of Textile Auxiliaries

St. Jost *Textil-Rund.*, 16 (July 1961) 390-398
The table lists trade names, manufacturers, composition and special advantages or uses of 126 agents. P.B.S.

Electrochemical Investigations of Ninhydrin: Formation and Stability Range of Two-electron Reduction Products

L. Holleck and O. Lehmann
Mh. Chem., 92 (July 1961) 499-511
One of the two unhydrated keto groups of ninhydrin (I) is found to be completely reduced during potentiostatic two-electron reduction. At pH > 4.5 the reduction product is in a tautomeric quinonoid condition, which at pH 4.5-8 loses a proton and 1 mol. H_2O , and at pH > 11 loses two protons. In contrast to the colourless benzenoid $2e^-$ reduction product, the simple ionised quinonoid cpd. is red and the doubly ionised quinone blue. The electro-neutral quinonoid cpd. below pH 4.5 is colourless and dehydrated. At pH > 9, a proton dissociates from the hydrated central keto group of I (pK = 8.6), and at higher pH values there is rapid transformation into α -carboxymandelic acid. I is two-electron-oxidised to α -carboxyphenylglyoxylic acid. H.H.H.

Auxiliaries for Prevention of Gas-fume Fading of Dyed Cellulose Acetate Fibres

E. P. Fricser *S.V.F. Fachorgan*, 16 (July 1961) 448-452
The causes, conditions for, and mechanism of gas-fume fading are explained. Methods of protection are discussed and the characteristics of the ideal gas-fading inhibitor listed. A brief survey of the patent literature on gas-fading inhibitors is given, together with a summary of methods of testing fastness to gas-fumes. P.B.S.

Compounds for Imparting Crease Resistance—the Urons

J. T. Marsh *Text. Rec.*, 78 (July 1961) 55-56
Derivatives of uron, which is a hypothetical compound of urea with four molecules of formaldehyde, are useful products for crease-resist finishing of rayon and cotton and are available in America under the trade name Dextraest. The nature of the chemical compounds has not been rigorously proved but, as the products are said to contain 30% of dimethylol urea, it is not surprising that there is some chlorine-retention damage on laundering. W.G.C.

Condensation of Formaldehyde with Perfluoroolefins—Tetrafluoroethylene, Hexafluoropropylene, and Trifluorochloroethylene

B. L. Dyatkin, E. P. Mochalina, and I. L. Knunyants
Doklady Akad. Nauk S.S.S.R., 139 (No. 1) (1 July 1961) 106-109

The condensation of paraformaldehyde with tetrafluoroethylene in presence of chlorosulphonic acid at 100°C produces α -difluorohydroacrylic acid and, with hexafluoropropylene at 130-150°C, α -fluoro- α -trifluoromethylhydroacrylic acid. In the case of trifluorochloroethylene, α -fluoro- α -chlorohydroacrylic acid and α -difluorohydroacrylic acid are obtained. The mechanism of these reactions is discussed. I.G.

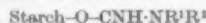
Polymethylene Polyphenols

W. R. Brookes *Ind. Eng. Chem.*, 53 (July 1961) 570-572
Improved resilience and flexibility of phenol-formaldehyde resins may be achieved by use of internal plasticisers, e.g. waxphenol, prepared by chlorination of paraffin and alkylation of phenol with the chlorinated deriv. Alkaline catalysis of waxphenol and formaldehyde produces methylol waxphenol which may be increased in mol. wt. by thermal processes to give a cured resin or, on reaction with further phenol and formaldehyde, form a cross-condensate monomer used as intermediate in preparation of moulding cpd. or laminating varnish. Improved flexibility, which is attributed to presence of polymethylene chains as an integral part of the structure, gives improved impact strength and, in the laminated varnishes, good cold-punching properties useful in preparation of printed electrical circuits. F.J.

PATENTS

Size for Cellulosic Materials

Corn Products Co. BP 874,020 (U.S.A. 16 Nov 1956)
Starch derivatives of formula



(R^1 and R^2 = Alk, subst. Alk, alkene, Ar or aralkyl) or one prepared by treating starch in presence of an alkaline catalyst with either an amine of formula $R^1\text{NR}^2\text{R}^3$ (R^3 = 2,3-epoxypropyl or 3-halogeno-2-hydroxypropyl; R^4 and R^5 = H, Alk, subst. Alk, alkene, Ar, aralkyl, 2,3-epoxypropyl or 3-halogeno-2-hydroxypropyl or N, R^4 and R^5 may together form a monoheterocyclic radical) or a quaternary salt having as cation $R^1\text{N}^+\text{R}^2\text{R}^3\text{R}^4$ (R^4 , R^5 and R^6 = Alk, subst. Alk, alkene, Ar, aralkyl, 2,3-epoxypropyl or 3-halogeno-2-hydroxypropyl, and if they are all the same then no one is of > 4 C) are substantive to cellulose and cellulose acetate. They enable sizing to be carried out with substantial exhaustion of the sizing liquor.

USP 2,965,518 (8 Oct 1958)

Cationic carbohydrates, e.g. modified starches of formula



(R^1 and R^2 = Alk, alkene, Ar and aralkyl) made by the process of USP 2,883,887 are picked up by cellulosic materials aliquot-wise with the aqueous medium. They have the additional advantage that they do not need pasting before the size liquor is made up. C.O.C.

Stable Aqueous Dispersions of Cellulose

American Viscose Corp.

BP 874,945 (U.S.A. 28 Jan 1957)

"Level-off D.P." cellulose (the crystalline cellulose obtained by Battista's method, *Ind. Eng. Chem.*, **42** (1950) 502) is mechanically disintegrated in an aqueous medium. The product applied to glass and dried yields a highly adherent continuous but readily removable film, e.g. on glass sheets or fibres.

C.O.C.

Antifoam Agents

Fabriques de Levure, Sucre & Alcools de Saint-Ouen-l'Aumône

BP 875,113 (France 27 Aug 1958)

An emulsion of either the oil-in-water or water-in-oil type consisting of 0.5–3.0% by wt. of an organopolysiloxane soluble in alcohol and oil, but insoluble in water, 20–30% of an oil or fat, 5–10% of an emulsifying agent, 1–4% of a stabiliser, the remainder being water, is a valuable antifoam agent.

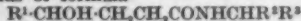
C.O.C.

Thickeners for Liquid Non-ionic Surfactants

General Aniline

USP 2,958,665 (24 July 1957)

Compounds of formula



($R^1 = H$ or CH_3 ; $R^2 =$ organic radical of 10–30 C free from water-solubilising groups; $R^3 = H$ or alkyl of 1–18 C; $R^2 + R^3$ contain 10–48 C), e.g. $OHCH_2-CH_2-CH_2-CONHCH_2-(CH_2)_8-CH_3$, obtained by treating *N*-decylamine with γ -butyrolactone, are useful gelling or thickening agents for liquid non-ionic surfactants.

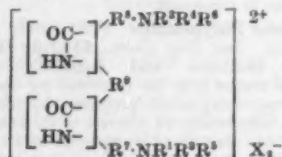
C.O.C.

Levelling Agents for Use in Dyeing Acrylic Fibres and Folds with Basic Dyes

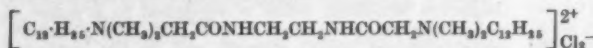
Ciba

BP 874,033 (Switzerland 14 Jan 1957)

Compounds of formula



(R^1 and $R^3 =$ aliphatic hydrocarbon of 10–18 C; R^2 , R^4 , R^5 and $R^6 =$ aliphatic hydrocarbon radical; R^7 , R^8 and $R^9 =$ alkylene; $X =$ anion), e.g.—



are excellent levelling and retarding agents for use in dyeing polyacrylonitrile with basic dyes.

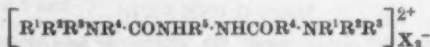
C.O.C.

Softening Agents for Cellulosic and Synthetic-polymer Textiles

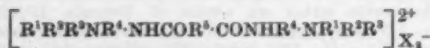
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BP 874,032 (Switzerland 14 Jan 1957)

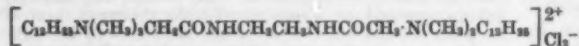
Compounds of formula



or



($R^1 =$ aliphatic hydrocarbon of 10–18 C; R^2 and $R^3 =$ aliphatic hydrocarbon; R^4 and $R^5 =$ alkylene; $X =$ anion), e.g.



are excellent softening agents for cotton, cellulose acetate, nylon, polyester fibres, and especially acrylic fibres.

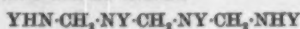
C.O.C.

Water- and Oil-repellent Agents

DuP

USP 2,958,613 (21 Jan 1959)

Treatment of compounds of formula $X(CF_3)_n-CH_2-O-CONH_2$ ($X = H$ or Hal ; $n = 7-12$) with $HCHO$ under strongly acid conditions yields a mixture of two principal components which can be readily separated by distillation. The first of these has the formula



($Y = X(CF_3)_n-CH_2-O-CO-$) and can be applied direct to textile fibres from organic-solvent solution or aqueous dispersion to produce an oil- and water-repellent finish which is fast to washing. The other component is $Y-NH-CH_2-NHY$, which is not suitable for application to textiles, but when treated with $HCHO$ under strongly acid conditions it is converted into the first component.

C.O.C.

Urea-Dialdehyde Starch Derivatives

Miles Laboratories

BP 875,542 (U.S.A. 6 Oct 1958)

2,3-Dialdehyde starch is treated for 1–24 h at 20–70°C with urea in presence of water and/or acetone and/or a lower alcohol. The resulting product is filtered off and dried. It is not soluble in alkali at normal temperature and is less soluble in dilute acids than the original oxystarch. It can be used to produce crease- and crush-proof textiles, as a size for paper, and as a moulding powder.

C.O.C.

Multi-component Vinylidene Interpolymer Latexes as Textile Finishes

Monsanto Chemical Co.

USP 2,959,497 (21 Oct 1957)

Aqueous latexes of interpolymers of (1) 25–60% by wt. of an alkanol acrylate or methacrylate (alkanol of 5–20 C with 5–14 C in the longest continuous chain), (2) 3–10% of acrylo- or methacrylo-nitrile, (3) 2–5% of acrylic, methacrylic, cinnamic, atropic, or crotonic acid, and (4) 60–25% of a monovinylidene aromatic hydrocarbon which may be chlorinated, are cheap and effective agents for imparting a soft, full handle and otherwise improving the physical properties of fabrics. Thus nylon taffeta is given a soft, full handle, cotton cloth is given good crease-recovery properties, and woollen fabric is rendered shrink-resistant by impregnation with such a latex.

C.O.C.

Aqueous Dispersion of Polypropylene

Montecatini Societa Generale per l'Industria Mineraria et Chimica

BP 874,173 (Italy 9 Oct 1956)

Aqueous dispersions, prepared in any convenient manner, of unbranched, head-to-tail polypropylene of crystalline content 50–100% (by wt.) are used for coating and impregnating a wide variety of substrates, from metals to textiles, e.g. to give impermeability to gases and water, protection against corrosion and chemical attack, as a laminating agent, and to improve the crease-recovery properties of textiles.

C.O.C.

Accelerators for Driers (V p. 576)

Rendering Permeable Materials Bacteriostatic (X p. 587)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS**Imidazole Dyes. IV—Condensation of o-Phenylenediamine with Phthalic Anhydride**

J. Arient and J. Marhan

Collection Czechoslov. Chem.

Commun., **26** (1961) 93–106 (in German)Chem. Abs., **55** (7 Aug 1961) 15488

Adding conc. HCl (9 ml) to *o*-phenylenediamine (I) (5.4 g) and phthalic anhydride (II) (7.4 g) suspended in methanol (50 ml), refluxing for 30 min with agitation and then cooling, yielded *N*-(*o*-aminophenyl)phthalimide hydrochloride (III) (8.5 g). Refluxing I (10.8 g), methanol (50 ml), II (7.4 g), and conc. HCl (6 ml) for 3 h, cooling and filtering, gave *o*-bis(2-benzimidazolyl)benzene (IV) (5.85 g); the filtrate contained III and 2-(*o*-carboxyphenyl)-benzimidazole (V). Refluxing I (5.4 g), II (7.4 g), water (50 ml), and aq. HCl (9 ml) for 3 h gave V (11.85 g), the same result being obtained using acetic acid, pyridine and nitrobenzene as solvents. Heating in a sealed tube I (1.1 g), II (1.5 g), and water (10 ml) for 1 h at 180–185°C gave IV (2.15 g); with 5–30% aq. HCl instead of water V (about 2.75 g) was obtained. Heating III (1 g) for 20 min at 250–280°C or V (1 g) for 20 min at 270–290°C gave 1,2-benzoylenebenzimidazole (VI) (0.65 g) and this was obtained also by refluxing III and V in acetic anhydride for 30 min. III was rearranged to V by refluxing in water, aq. HCl , pyridine, acetic acid, and nitrobenzene, whereas refluxing III in methanol resulted in no change.

C.O.C.

Dyes with Antipyrine Nuclei. VII—Effect of Substituents on Colour and Acid-Basic Properties of Dyes

O. F. Ginzburg and De Chun-Sen

Zhur. obshch. khim., 31 (Apr 1961) 1219-1222

Amino and diethylamino groups have been introduced *para* to the central C atom of antipyrine (1,5-dimethyl-2-phenyl-3-pyrazolone) dyes. Absorption spectra and acid-basic properties of compounds obtained have been examined. With increasing nucleophilic nature of the amino group the hue of the dyes deepens and the resistance to hydrolysis increases. With other substituents, e.g. NO_2 , the deepening of hue is not accompanied by an increase in resistance to hydrolysis. I.G.

Synthesis of Azoferrocene, its Reduction and Behaviour in Conditions of Benzidine Regrouping

A. N. Nesmeyanov, E. G. Perevalova, and T. V. Nikitina

Doklady Akad. Nauk S.S.S.R., 138 (No. 5) (11 June 1961) 1118-1121

Azoferrrocene has been prepared by reacting nitrous oxide with ferrocene-lithium. Benzidine compounds are not formed when azoferrrocene is treated with acids, in contrast to the case of azobenzene. The different behaviour is probably due to the presence of the Fe^0 atom. Reduction of azoferrrocene produces ferrocenylamine only; the hydrazo compound is formed as an intermediate, rapidly disappearing product, while the reduction of azobenzene produces, quantitatively, hydrazobenzene and no amine. I.G.

Reactive Dyes for Cellulose

I. D. Rattee

Endeavour, 20 (July 1961) 154-161

A review of the basic principles of dyeing, early research on dyes capable of reacting chemically with fibres, the development of practical esterifying (Procion, Procion H, Cibacron, Reactone, Drimarene) and etherifying (Remazol) dyes, reaction kinetics of dyeing with reactive dyes, and their limitations. M.T.

Development of the Drimarene Class of Reactive Dyes

M. Capponi, E. Metzger, and A. Giamara

Amer. Dyestuff Rep., 50 (10 July 1961) 505-514

The introduction of reactive dyes based on cyanuric chloride led to a search for other reactive systems as a basis for dyes. The Drimarene Z reactive dyes, based on tetrachloropyrimidine, are primarily intended for printing techniques and the related padding processes on cellulosic fibres. In their development no attempt was made to produce all-round reactive dyes which could be recommended for every possible type of application. They may, however, also be applied to wool unions and silk by the cotton method, after treating the wool union with a cationic product if the dyeing is not originally of adequate fastness, and to polyamide fibres in the presence of acid or an acid-yielding agent. S.B.D.

Procynyl Dyes—New Reactive Dyes for Nylon

Tintoria, 58 (May 1961) 207-209

Summary of a conference held at the Istituto Nazionale de Sefificio on 26 Feb 1961. There are graphs showing the exhaustion and dye fixation *v.* pH for Procynyl Blue RS (C.I. Reactive Blue 6) (I) and Procynyl Scarlet GS (C.I. Reactive Red 10) (II); *v.* temp. for I; exhaustion *v.* liquor ratio for I, Procynyl Yellow GS (C.I. Reactive Yellow 5) and Procynyl Orange GS (C.I. Reactive Orange 3) (III); dye fixed *v.* time for II and III. All experiments were carried out with nylon 6.6. E.V.T.

Effect of Spatial Factors on the Properties of Dyes Containing the Biphenyl Radical

XVI—Monoazo Dyes, Derivatives of Benzene, Diphenyl and *p*-Terphenyl containing Amide Groups

B. M. Krasovitskii, B. I. Ostrovskaya, and N. I. Titarenko

Ukrain. khim. zhur., 27 (No. 2) (1961) 226-230

Dyeing of cotton with dyes containing amide groups and varying numbers of benzene rings in the molecule has been investigated. An increase in the number of benzene rings increases the affinity for cotton, while the effect of structural differences in isomeric dyes is very slight. The colour of aqueous solutions of the dyes is most intense when the azo group is combined directly with the diphenyl ring.

XVII—Monoazo Dye from 4''-substituted Derivatives of 4-Amino-*p*-terphenyl

B. M. Krasovitskii and N. I. Titarenko *Ibid.*, 230-234

A series of monoazo dyes has been obtained by reacting diazotised derivatives of 4-amino-*p*-terphenyl with H acid in an alkaline medium. The affinity of cotton for these dyes increases with increasing number of benzene rings in the molecule. The presence of an amino group in the 4''-position increases the affinity to a higher degree than does the presence of a hydroxy or methoxy group. The colour of aqueous solutions of these dyes is not affected by the substituents in this position.

XVIII—Non-symmetrical Disazo Dyes from 4,4''-Diamino-*p*-terphenyl

B. M. Krasovitskii and N. I. Titarenko

Ibid., 27 (No. 3) (1961) 390-395

The substantivities of non-symmetrical dyes obtained from 4,4''-diamino-*p*-terphenyl are higher than those of similar, non-symmetrical benzidine dyes. In addition, the colour is displaced towards the yellow. I.G.

Reduction of Brilliant Black BN (C.I. Food Black 1) with Formation of Monoazo Dyes

I. S. L. Ruiz and C. Laroche

Ann. fals. et expert. chim., 53 (1960) 581-592

Chem. Abs., 55 (24 July 1961) 14742

Analyses of confectionery containing the above dye consistently showed presence of two orange dyes, di-Na 4(sulphophenylazo)-1-amino-7-sulphonaphthalene and di-Na 4(sulphophenylazo)-1-hydroxy-7-sulphonaphthalene, which are not normal food additives. The first of these is produced from Brilliant Black BN by heating with a reducing sugar and it, in turn, is converted into the second orange dye during the analysis, where an extraction referred to as double staining on wool takes place. C.O.C.

Replacement of Halogen in Azo Compounds. XIII—Replacement of Chlorine by Alkoxy and Aroxy Groups in Disazo Dyes obtained from 3,3'-Dichlorobenzidine and the Preparation of Ethers of 4,4'-Diamino-3,3'-dihydroxydiphenyl

N. A. Rozanell'akaya and B. I. Stepanov

Zhur. obshch. khim., 31 (No. 3) (Mar 1961) 758-764

Interaction of 4,4'-bis(2''-hydroxy-5''-methylbenzeno-1''-azo)-3,3'-dichlorodiphenyl(disazo dye obtained from 3,3'-dichlorobenzidine and *p*-cresol) with sodium alkoxides and phenoxides (NaOR) in presence of copper acetate brings about replacement of both chlorine atoms by alkoxy and aroxy groups; $\text{R} = \text{C}_2\text{H}_5$; $\text{n-C}_4\text{H}_9$; $(\text{CH}_3)_2\text{CH}$; $(\text{CH}_3)_3\text{C}$; $\text{n-C}_6\text{H}_{13}$; $\text{n-C}_8\text{H}_{17}$; $\text{n-C}_{12}\text{H}_{25}$; C_6H_5 ; $(\text{CH}_3)_2\text{CH}$; $2\text{-CH}_2\text{C}_6\text{H}_4$; $4\text{-CH}_2\text{C}_6\text{H}_4$; $3,5\text{-(CH}_3)_2\text{C}_6\text{H}_3$. Thus 11 disazo dyes (brown to red) have been prepared which, in contrast to monoazo dyes, do not dye polyamide fibre by the suspension method. It is found that, in the presence of a copper salt, the mobility of chlorine in these azo cpd. is comparable to that in other *o*-chloro-*o'*-hydroxyazo cpd. Reductive cleavage (SnCl_2 followed by sodium hydro-sulphite) of these dyes yields 11 ethers of 4,4'-diamino-3,3'-dihydroxydiphenyl. G.J.K.

Reactive Methylene Compounds. VIII—Condensations with Benzenediazonium Salts

H. G. Garg and S. N. Mehra

J. Indian Chem. Soc., 38 (May 1961) 325-326

The condensation products of 4-methoxy-, 2-, 3-, and 4-chloro-, 3- and 4-nitro-, 4-methyl-, 2-chloro-4-nitro-, and 4-chloro-2-nitro-benzene diazonium chlorides, with diethyl malonate, 1-carbamoyl-3-methyl-5-pyrazolone, indan-1,3-dione, and methone are described. It has been observed that 1-carbamoyl-3-methyl-4-(substituted benzenazo)-5-pyrazolones lose the carbamoyl group when heated in alcohol to form 3-methyl-4-(substituted benzenazo)-5-pyrazolones. H.H.H.

Acid Dye from 2-Methyl-1-naphthylamine-6-sulphonic Acid

W. Wojtkiewicz and J. Szadowski

Zesz. Nauk. Politech. Łódz., No. 33

(*Chemia* 9) (1961) 113-119

2-Methyl-1-naphthylamine-6-sulphonic acid was prepared from 2-methylnaphthalene, a 25-4% yield of pure product and 40% of lesser quality acid being obtained. The acid can be diazotised and coupled in alk. medium with β -naphthol to give a yellowish red acid dye. It is assumed that the presence of a CH_3 group *ortho* to the azo groups causes a hypsochromic effect. T.Z.W.

Studies on Rapidogen Dyes. II—Diazoamino Compounds Synthesised by using *N*-Methylantranilic Acid, Guanylurea, and Amino-G-Acid as Stabilisers

R. Asaoka and T. Sekiguchi

Rept. Govt. Chem. Ind. Research Inst. Tokyo, 56 (Feb 1961) 22-26

Twelve kinds of diazoamino compounds were prepared by using 4-nitro-*o*-toluidine (Fast Scarlet G Base), 2,5-dichloroaniline (Fast Scarlet GG Base), 4-chloro-*o*-anisidine (Fast Red R Base), and 5-chloro-*o*-toluidine (Fast Red TR Base), as diazo components, and the title cpd. as stabilisers. (*Abstractor's Note*—The Colour Index states that Fast Scarlet G Base (C.I. Azoic Diazo Component 12) is 5-nitro-*o*-toluidine; Fast Red TR Base (C.I. Azoic Diazo Component 11) is 4-chloro-*o*-toluidine hydrochloride.) When Fast Red R Base was used with guanylurea, the diazoamino cpd. was obtained in good yield. Amino-G-acid gave diazoamino cpd. of high aqueous solubility and Fast Scarlet GG Base gave ones of high stability. M.T.

1,4-Naphthoquinone Dyes

A. Inoue, N. Kuraki, and K. Konishi

Bull. Univ. Osaka Prefecture, Ser. A8, No. 1 (1959) 31-55
Chem. Abs., 55 (10 July 1961) 13853

1,4-Naphthoquinone (I) was converted to 1,4-dimethoxy- and 1,4-diethoxy-naphthalenes (II); mononitration and reduction gave 2-amino-1,4-dialkoxynaphthalenes (III), which were diazotised and coupled to Naphtol AS, AS-OL, and AS-8W (C.I. Azoic Coupling Component 2, 20 and 7). III condensed with BON acid gave amides (IV), developed on cotton with Scarlet G and Scarlet GG (C.I. Diazo Component 12 and 3). 2,3-Dichloro derivatives of I and II were synthesised, and dyes obtained by coupling 3-chloro-2-amino-II to Naphtols AS, AS-BG (C.I. Azoic Coupling Component 19), amides IV and BON acid 2,5-dimethoxyaniline. I was converted to 1,2,4-trihydroxynaphthalene, thence to 2-hydroxy-I, which were condensed with *o*-phenylenediamine to give 6-hydroxynaphthophenazine, used as coupling component in a series of dyes. 2,3-Dichloro- and 5-nitro-2,3-dichloro-I were condensed with amines by dehydrochlorination, and the position *ortho* to the imino group was substituted by Br, dehalogenated, and cyclised to carbazole vat dyes. Air blown into a mixture of I, copper acetate, and an amine A gave 2-A-NH-I, some of which were disperse dyes, or Y-A-Y (Y = 1,4-naphthoquinon-2-ylamino). I with Sn and the appropriate alcohol in presence of HCl gave 4-alkoxy-1-naphthols, used as coupling components in a series of dyes. Also described are 3,5(or 8)-disubst. 2-hydroxy-I, 3,5(or 8)-disubst.-1-phenylazo- and 1-(2,4-dinitrophenylazo)-2,4-dihydroxynaphthalenes. A.T.P.

Direct Introduction of the Diazonium Group into the Pyrazole Nucleus

H. P. Patel, J. M. Tedder, and B. Webster

Chem. and Ind., No. 30 (29 July 1961) 1163

Direct introduction of the diazonium group into the nucleus of 3,5-dimethylpyrazole has been achieved. The latter cpd. (3.6 g) was dissolved in 75 ml water containing 26 g sodium nitrite. 188 ml of 2-N HCl were added and the mixture maintained at 5°C for 20 h, after which a further 95 ml of 2-N HCl were added. On standing for 24 h, excess nitrite was removed with sulphamic acid and the soln. made alkaline. Addition of 200 g NaCl, followed by extraction with chloroform, yielded 3.3 g crude 4-diazo-3,5-dimethylpyrazole. Recrystallisation from benzene gave a product, λ_{max} 475 m μ , m.p. 65°C, which could be coupled with β -naphthol in boiling chloroform or, under aq. alkaline conditions, after conversion to the diazonium salt. F.J.

Preparation of 3-Hydroxythionaphthene Dyes

M. Lipp and S. M. Abd Elrahman Omran

Melliand Textilber., 42 (July 1961) 792-795

The coupling of 3-hydroxythionaphthene with diazonium salts is discussed. The properties of 21 of the azo compounds are listed in three tables which give—m.p., elemental analysis, light fastness, light absorption, and a synopsis of the infrared spectra. P.B.S.

Investigations on Synthetic Dyes

XVIII—Synthesis of Isomeric Quaternary Salts of *N*-Arylquinaldine and their Reactions

G. T. Pilyugin and E. P. Opanasenko

Zhur. obshch. Khim., 31 (Apr 1961) 1233-1240

1-*N*-methoxyphenylquinaldine and 1-phenyl-6-methoxyquinaldine were prepared by condensation of

N-methoxyphenylamine with paraldehyde or vinylbutyl ether. Perchlorate salts of these isomers were prepared and condensed with various compounds (quinoline iodo-methylate, methylbenzothiazole iodoethylate, *N*-dimethylaminobenzaldehyde, etc.) to obtain cyanine dyes of various compositions. The colour of the dyes containing the methoxy group in position 6 of the quinoline ring is more intense than that of their *N*-isomers.

XXI—Styryl Dyes obtained from *N*-Arylquin-aldine Quaternary Salts

G. T. Pilyugin and I. N. Chernyuk

Ibid., 31 (Apr 1961) 1240-1244

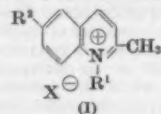
Seven new styryl dyes have been obtained by condensing quaternary salts of *N*-arylquinaldine with *N*-acetylaminobenzaldehyde. Absorption spectra in the visible region have been determined. A hypsochromic shift of the absorption maxima of about 100 m μ is produced by acetylation of the amino group. I.G.

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G. T. Pilyugin and I. N. Chernyuk

Ibid., 31 (May 1961) 1585-1587

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($R^1 = C_6H_5$, $C_6H_4CH_3$ -*p*, or $C_6H_3H_2$ - α and $-\beta$; $R^2 = H$, or CH_3 ; and also the 5,6-benzo deriv. of I where $R^2 = H$; $X = ClO_4$ or iodide) with *p*-hydroxybenzaldehyde gave 6 previously undescribed dyes. The absorption max. of I undergo a hypsochromic shift (~ 90 -100 m μ) compared with the corresponding *p*-amino- and *p*-dimethylaminostyrenes, and a bathochromic displacement (18-20 m μ) compared with the acetyl-amino-substituted analogues. G.J.K.

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Studies on Rapidogen Dyes. II—Diazamino Compounds Synthesised by using N-Methylanthranilic Acid, Guanylurea, and Amino-G-Acid as Stabilisers R. Asaka and T. Sekiguchi

Rept. Govt. Chem. Ind. Research Inst. Tokyo, 56 (Feb 1961) 22-28

Twelve kinds of diazomino compounds were prepared by using 4-nitro-*o*-toluidine (Fast Scarlet G Base), 2,5-dichloroaniline (Fast Scarlet GG Base), 4-chloro-*o*-anisidine (Fast Red R Base), and 5-chloro-*o*-toluidine (Fast Red TR Base), as diazo components, and the title cpd. as stabilisers. (*Abstractor's Note*—The Colour Index states that Fast Scarlet G Base (C.I. Azoic Diazo Component 12) is 5-nitro-*o*-toluidine; Fast Red TR Base (C.I. Azoic Diazo Component 11) is 4-chloro-*o*-toluidine hydrochloride.) When Fast Red R Base was used with guanylurea, the diazomino cpd. was obtained in good yield. Amino-G-acid gave diazomino cpd. of high aqueous solubility and Fast Scarlet GG Base gave ones of high stability. M.T.

1,4-Naphthoquinone Dyes

A. Inoue, N. Kuraki, and K. Konishi

Bull. Univ. Osaka Prefecture, Ser. A8, No. 1 (1959) 31-55
Chem. Abs., 55 (10 July 1961) 13853

1,4-Naphthoquinone (I) was converted to 1,4-dimethoxy- and 1,4-diethoxy-naphthalenes (II); mononitration and reduction gave 2-amino-1,4-dialkoxynaphthalenes (III), which were diazotised and coupled to Naphthol AS, AS-OL, and AS-SW (C.I. Azoic Coupling Component 2, 20 and 7). III condensed with BON acid gave amides (IV), developed on cotton with Scarlet G and Scarlet GG (C.I. Diazo Component 12 and 3). 2,3-Dichloro derivatives of I and II were synthesised, and dyes obtained by coupling 3-chloro-2-amino-II to Naphthols AS, AS-BG (C.I. Azoic Coupling Component 19), amides IV and BON acid 2,5-dimethoxyaniline. I was converted to 1,2,4-trihydroxynaphthalene, thence to 2-hydroxy-I, which were condensed with *o*-phenylenediamine to give 6-hydroxynaphthophenazine, used as coupling component in a series of dyes. 2,3-Dichloro- and 5-nitro-2,3-dichloro-I were condensed with amines by dehydrochlorination, and the position *ortho* to the imino group was substituted by Br, dehalogenated, and cyclised to carbazole vat dyes. Air blown into a mixture of I, copper acetate, and an amine A gave 2-A-NH-I, some of which were disperse dyes, or Y-A-Y (Y = 1,4-naphthoquinone-2-ylamino). I with Sn and the appropriate alcohol in presence of HCl gave 4-alkoxy-1-naphthols, used as coupling components in a series of dyes. Also described are 3,5(or 8)-disubst. 2-hydroxy-I, 3,5(or 8)-disubst.-1-phenylazo- and 1-(2,4-dinitrophenylazo)-2,4-dihydroxynaphthalenes. A.T.P.

Direct Introduction of the Diazonium Group into the Pyrazole Nucleus

H. P. Patel, J. M. Tedder, and B. Webster

Chem. and Ind., No. 30 (29 July 1961) 1163

Direct introduction of the diazonium group into the nucleus of 3,5-dimethylpyrazole has been achieved. The latter cpd. (3.6 g) was dissolved in 75 ml water containing 26 g sodium nitrite. 188 ml of 2-N HCl were added and the mixture maintained at 5°C for 20 h, after which a further 95 ml of 2-N HCl were added. On standing for 24 h, excess nitrite was removed with sulphamic acid and the soln. made alkaline. Addition of 200 g NaCl, followed by extraction with chloroform, yielded 3.3 g crude 4-diazo-3,5-dimethylpyrazole. Recrystallisation from benzene gave a product, λ_{max} 475 m μ , m.p. 65°C, which could be coupled with β -naphthol in boiling chloroform or, under aq. alkaline conditions, after conversion to the diazonium salt. F.J.

Preparation of 3-Hydroxythionaphthene Dyes

M. Lipp and S. M. Abd Elrahman Omran

Melliand Textilber., 42 (July 1961) 792-795

The coupling of 3-hydroxythionaphthene with diazonium salts is discussed. The properties of 21 of the azo compounds are listed in three tables which give—m.p., elemental analysis, light fastness, light absorption, and a synopsis of the infrared spectra. P.B.S.

Investigations on Synthetic Dyes

XVIII—Synthesis of Isomeric Quaternary Salts of N-Arylquinaldine and their Reactions

G. T. Pilyugin and E. P. Opanasenko

Zhur. obshch. khim., 31 (Apr 1961) 1233-1240

1-N-methoxyphenylquinaldine and 1-phenyl-6-methoxyquinaldine were prepared by condensation of

N-methoxyphenylamine with paraldehyde or vinylbutyl ether. Perchlorate salts of these isomers were prepared and condensed with various compounds (quinoline iodo-methylate, methylbenzothiazole iodoethylate, N-dimethylaminobenzaldehyde, etc.) to obtain cyanine dyes of various compositions. The colour of the dyes containing the methoxy group in position 6 of the quinoline ring is more intense than that of their N-isomers.

XXI—Styryl Dyes obtained from N-Arylquinaldine Quaternary Salts

G. T. Pilyugin and I. N. Chernyuk

Ibid., 31 (Apr 1961) 1240-1244

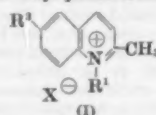
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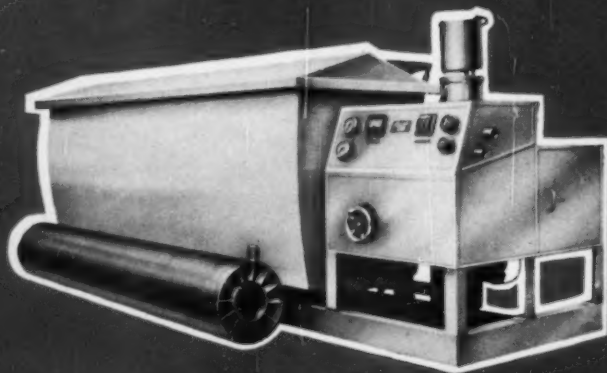
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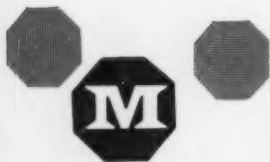
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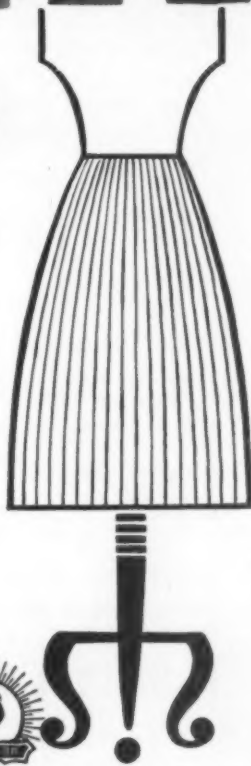
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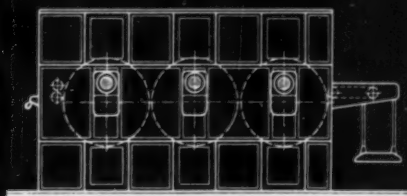
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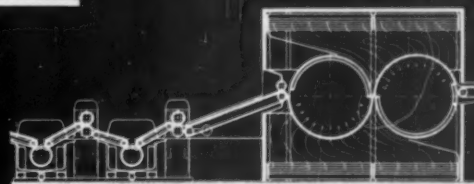
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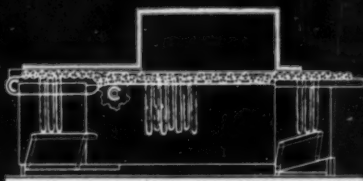
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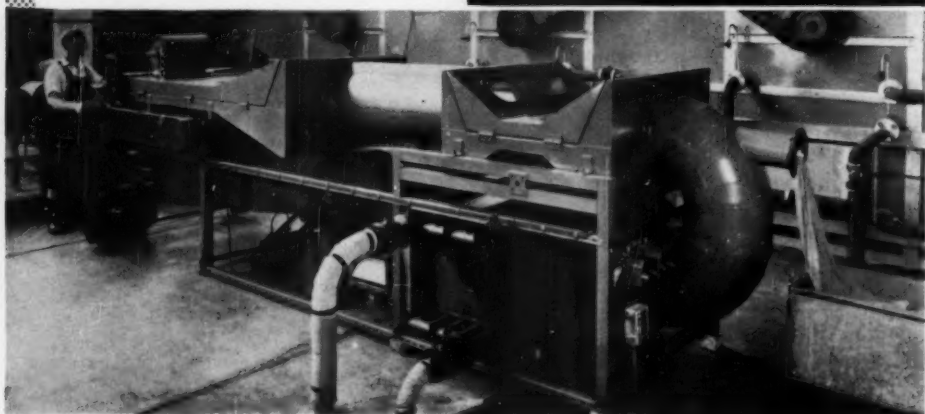
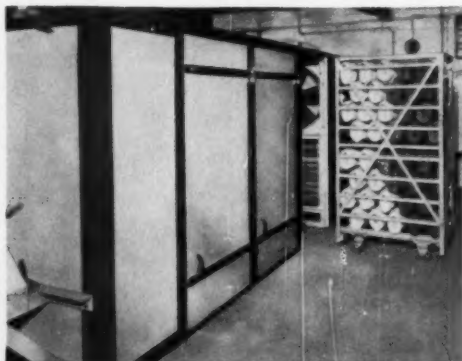
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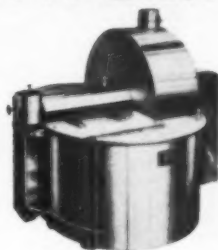
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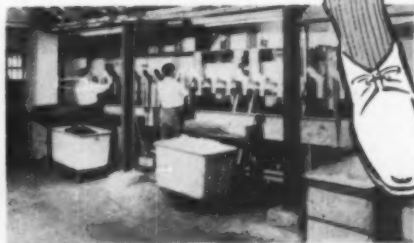


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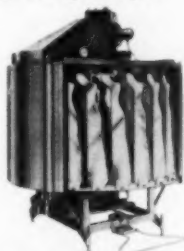
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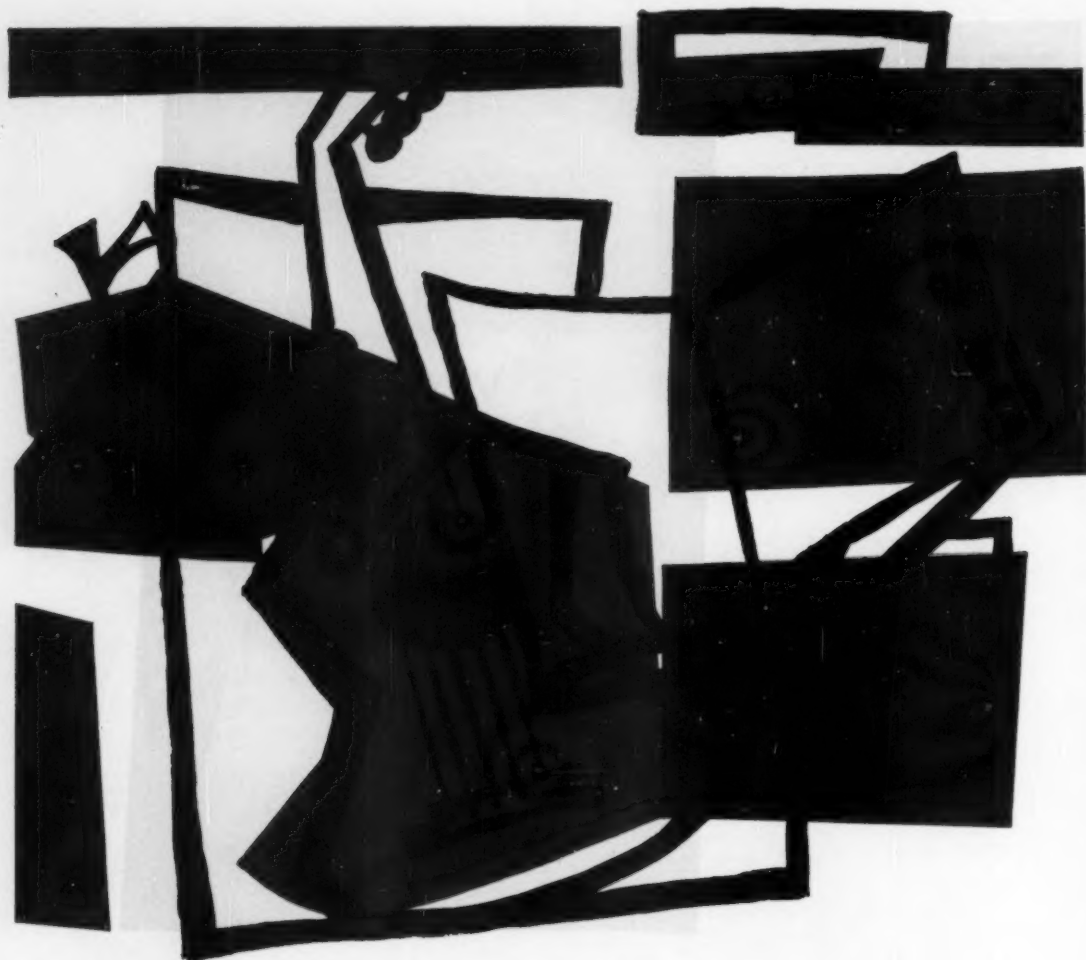


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113 Sterling Road, Toronto, 3.

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11 Nassau Street, Dublin.



with phenols, but *N*-methyl-9-hydrazoneacridanone is too sensitive to oxidation. Alternative syntheses were therefore explored, and preparations for many derivatives are described. E.S.

Colour and Constitution. VII—Diazamerocyanines

S. Hünig and H. Herrmann

Ibid., 32-41

Chem. Abs., *ibid.*, 12859

An examination and discussion of the absorption spectra of the dyes of the previous abstract. E.S.

Cyanine Dyes, Derivatives of 2-Methylantraquinone-(1',2':5,4)-imidazole

A. V. Stetsenko and G. V. Chernyavskii

Ukrain. khim. zhur., 27 (No. 2) (1961) 237-240

Quaternary salts of 2-methyl-1-*p*-tolylantraquinone-(1',2':5,4)-imidazole, prepared by reacting 1-chloro-2-acetylaminooanthraquinone with *p*-toluidine in isoamyl alcohol in presence of copper acetate have been obtained and used for the synthesis of 7 cyanine dyes. Absorption spectra of these dyes indicate that electronegative substituents in the benzimidazole group of symmetrical cyanine dyes produce greater intensity of colour. I.G.

Cyanine Dyes from Seven-membered Heterocyclic Systems. III—Merocyanines and Rhodacyanines in the Series of Tetrahydrohepta-1,4-thiazine and Tetrahydrohepta-1,4-diazine

L. K. Mushkalo and Z. I. Shokol

Ukrain. khim. zhur., 27 (No. 3) (1961) 372-379

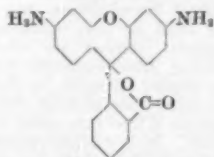
Ten new orange-red merocyanine dyes were prepared by fusing acetanilidomethylene derivatives of rhodamine with the bases of tetrahydrohepta-1,4-thiazine and tetrahydrohepta-1,4-diazine derivatives. Replacement of the sulphur atom in the seven-membered ring by NH has little effect on the colour of merocyanine dyes. Methyl groups in the β -position intensify the colour. Eight new rhodacyanine dyes were obtained by heating in pyridine equimolecular amounts of quaternary salts of merocyanines with quaternary salts of 2-methylbenzothiazole or of 4,5-diphenylthiazole. Absorption spectra of dyes obtained show, in addition to the band in the visible spectrum, an intense short-wave band with a maximum at 355-365 m μ . I.G.

Rhodamine Dyes and Related Compounds. I—The Parent Substance Rhodamine (C.I. 45170), its Preparation and Properties

I. S. Ioffe and V. F. Otten

Zhur. obshch. khim., 31 (May 1961) 1511-1516

The rhodamine dye obtained by condensation of *m*-aminophenol with phthalic anhydride is in fact a mixture of three dyes which are not separable by crystallisation from water. The pure dye (I) has been separated from the other two unspecified substances by paper chromatography and its structure confirmed by analysis and its conversion to fluorescein. In contrast to literature data, I is insoluble in alkali but with strong acids it forms monoacidic salts. In neutral and weakly alkaline medium I behaves as a dipolar ion which in acid medium is converted to a complex cation without changing its electronic structure. The spectral characteristics (400-560 m μ) of I do not change over the pH range 0-14. The electronic structure of I is disturbed through action of conc. acids or bases with the formation in the former case of the above coloured cation of a diacidic salt and in the latter case of a colourless anion, presumably a corresponding carbinol deriv. Acetylation of I yields a colourless lactone



G.J.K.

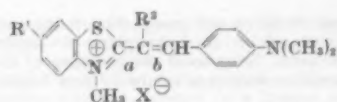
Steric Hindrance in Conjugated Chains of Polymethine Dyes

A. I. Kiprianov and F. A. Mikhailenko

I—Quaternary Salts of 2-(*p*-dimethylamino-styryl)-benzothiazoles

Zhur. obshch. khim., 31 (Mar 1961) 781-786

The bond of a in

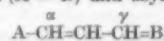


where $R^1 = H, NO_2$, or $N(CH_3)_2$; $R^2 = H$ or CH_3 ; $X = I$ affects the position of the absorption max. when $R^2 = CH_3$. For a single bond the bathochromic shift is strongest when $R^1 = N(CH_3)_2$ and least when $R^1 = NO_2$.

II—Symmetrical and Asymmetrical Carbo-cyanines

Ibid., 786-793

Symmetrical (A = B) and asymmetrical carbo-cyanines



A and B = *N*-methylquinoline, *N*-methyl-5-nitrobenzothiazole or *N*-methyl-5-dimethylaminobenzothiazole, have been investigated spectrophotometrically in the visible range. It is shown that the replacement of H by CH_3 in α and γ positions gives rise to steric hindrance causing hypso- or batho-chromic displacement of the absorption max. depending on the order of the bond around which rotation of A or B occurs. The order of the bond may be modified by changing the basicity of A and B.

G.J.K.

Photoreduction and Demethylation of Thiazine Dyes on Silica Sol

Kh. L. Arvan, G. A. Korsunovskii, and S. Yu. Lebedev

Doklady Akad. Nauk S.S.S.R., 139 (No. 2) (11 July 1961) 402-405

Thiazine dyes undergo a spectral shift when irradiated with visible light in presence of silica sol. In absence of air, thiazine dyes are reduced to their leuco compounds and this process is accompanied by demethylation. The reoxidation of leuco compounds produces a different dye with a corresponding spectral shift. It may be assumed that the dye forms adsorption bonds with surface OH groups of silica sol. The photoreduction of Methylene Blue (C.I. 52015) and other dyes containing methyl groups by hydrogen atoms of OH groups in silica sol must be accompanied by the replacement of methyl groups by hydrogen atoms and the methylation of surface OH groups in silica sol. I.G.

Structure of Complex Compound of Gallium Chloride and Methylene Blue

K. A. Bol'shakov, Yu. G. Eremin, and R. P. Evstigneeva

Izvest. Akad. Nauk S.S.S.R., oddel. khim. nauk, No. 5 (May 1961) 745-749

The presence of bonds between gallium and sulphur and nitrogen atoms in the complex compound of gallium chloride and Methylene Blue (C.I. 52015) has been ascertained by infrared analysis. The existence of such bonds, where the co-ordinate number of gallium is 6, has been confirmed by a study of the structure of Ga diethyldithiocarbamate and by potentiometric titration of the complex with NaOH. I.G.

Mechanism of a New Synthesis of 2-Chloroanthraquinone

G. Eigenmann, H. R. Rickenbacher, and H. Zollinger

Helv. chim. Acta, 44 (1 Aug 1961) 1211-1221

A mechanism for the thermal decomp. of 2,3,4a,9a-tetrahalogeno-1,2,3,4,4a,9a-hexahydroanthraquinones is presented which accounts for the production of mainly anthraquinone (I) in the decomp. of the tetrabromo-deriv., of mainly 2-chloroanthraquinone (II) from the tetrachloro-deriv., and of mixtures of I and II from the thermolysis of mixed dibromo-dichloro-hexahydroanthraquinones. H.H.H.

Separation Components in the Synthesis of Organic Dyes. XIII—The Behaviour of the Anthraquinone Nucleus

I. Reichel and W. Schmidt

Rev. chim. Acad. rep. populaire Roumaine, 5, No. 1 (1960) 107-117 (in German)

Acad. rep. populaire, Romine, Studii cercetari chim., 8 (1960) 213-226

Chem. Abs., 55 (20 May 1961) 10898

Sym. disazo dyes were prepared by tetrazotising 1,4- and 1,5-diaminoanthraquinone and coupling with phenol, acetacetanilide, 1-amino-8-naphthol-4,6-disulphonic acid, etc., and unsym. ones from 1-amino-5-oxalylaminoanthraquinone by diazotising, coupling with e.g. phenol,

hydrolysing off the oxalyl group, diazotising and coupling with 1-amino-2-*p*-nitrophenylazo-8-naphthol-3,6-disulphonic acid. In general the presence of the anthraquinone nucleus decreases solubility and brightness.

E.S.

Behaviour of Phthalocyanines in Aqueous Solution—I

K. Bernauer and S. Fallab

Helv. chim. Acta, 44 (1 Aug 1961) 1287-1292

Metal-free phthalocyanine-tetra-sulphonate and its Co, Mn, and Cu deriv. are found to dimerise in dilute aq. soln. Stabilities of these dimers, as determined by spectrophotometric measurements in water and 50% aq. ethanol, indicate a maximum stability for the metal-free cpd.

H.H.H.

Syntheses of Two Kinds of Metal-free Tetra-azaporphines by the Urea-melt Method

N. Fukada

Bull. Chem. Soc. Japan, 34 (June 1961) 884-885 (in English)

Mixtures of triammonium 4-sulphophthalate or quinolinic acid and urea, when heated at 170-250°C for 0.5-1.5 h with addition of boric acid and ammonium molybdate as catalysts, afforded ammonium phthalocyanine tetra-(4)-sulphonate or tetra-2,3-pyridinoporphyrazine in yields of 8-18%, increased to 26-42% by further addition of a powdered metal, e.g. Cr, Mo, or Mn. A small amount of metal chelate by-product was formed when Cr or Mn were used, but not with Mo.

H.H.H.

Orcein Pigments. XII—Syntheses of α -Hydroxy-orcein

H. Musso and H. Beecken *Chem. Ber.*, 94 (1961) 585-600

Orcein and Litmus (C.I. Natural Red 28) Pigments: Constitutional Elucidation and Constitutional Proof by Synthesis

H. Musso

Planta Med., 8 (1960) 432-446*Chem. Abs.*, 55 (7 Aug 1961) 15627

Orcein can be chromatographically separated into 14 nitrogenous compounds. The structure of the main components has been elucidated by use of resonance and electronic spectra methods. The chief pigments can be divided into 3 groups according to the nature of the chromophoric system: 7-hydroxyphenoxazine (I), 7-aminophenoxazine, and 7-aminophenoxazine. Litmus is a mixture of more complex pigments than orcein. Most components of litmus are polymers having 1 chromophores.

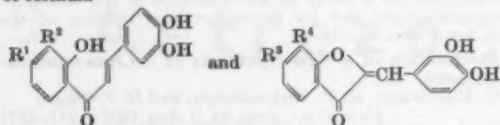
C.O.C.

Anthochlor Pigments. XIV—Pigments of *Vigniera multiflora* and *Baeria chrysostoma*

M. Shimokoriyama and T. A. Geissman

J. Org. Chem., 25 (1960) 1956-1959*Chem. Abs.*, 55 (7 Aug 1961) 15451

Among the mixture of pigments obtained were several of formula



They included one in which $R^1 = OH$ and $R^2 = H$, sulfuretin ($R^2 = OH$, $R^4 = H$) and sulfurein ($R^2 = O$ -glucosyl, $R^4 = H$).

C.O.C.

Anthocyanines. XXXIII—Delphin, the Anthocyanine of *Carcus sativus*

N. Saito, S. Mitsui, and K. Hayashi

Proc. Japan Acad., 36 (1960) 340-345 (in English)*Chem. Abs.*, 55 (7 Aug 1961) 15476

Evidence is presented to show that delphin is a 3,5-dimethoxydelphinidin (cf. C.I. 75190).

C.O.C.

Enzymic Decolorisation of Anthocyanine Pigments

D. M. Scheiner

Univ. Microfilms (Ann Arbor, Mich.),*L.C. Card No. Mic 61-548*, 69 pp.*Dissertation Abs.*, 21 (1961) 2882*Chem. Abs.*, 55 (7 Aug 1961) 15556

Structures of Phytoene, Phytofluene, ζ -Carotene, and Neurosporene

J. B. Davis, L. M. Jackman, P. T. Siddons, and B. C. L. Weedon

Proc. Chem. Soc., (July 1961) 261-263

The structures of the title cpd. (which are probably intermediates in the biosynthesis of lycopene and other

carotenoids) proposed by Rabourn and Quackenbush (cf. *Arch. Biochem. Biophys.*, 61 (1956) 111; *abs. Papers Amer. Chem. Soc.* (Sept 1957) 88C) are now confirmed by nuclear magnetic resonance spectra; their biosynthetic relationship with neurosporene is established.

H.H.H.

Mechanism of Leafing of Aluminium Bronzes (C.I. 77000)

B. N. Tyutyunnikov and Z. I. Bukhahtab

Trudy Khar'kov. Politekh. Inst. im. V. I. Lenina,*Ser. Khim. Tekhnol.*, 26 (6) (1959) 155-159*Chem. Abs.*, 55 (24 July 1961) 14935

Presence of a layer of stearic acid-Al stearate on the surface of the pigment particles is a prerequisite for leafing. This layer reduces the wettability of the pigment by the vehicle. This reduction in wettability is caused not by the layer proper but by the gases, mainly O_2 , in it. O_2 is highly soluble in the stearic acid-Al stearate layer and thus this layer acts as an oxygen-storing medium. Presence of Al stearate decreases the solubility of the layer in the vehicle and so prevents its removal. The need to cure polished Al bronze powder might be eliminated if the polishing were done in a stearic acid-Al stearate medium rather than in stearic acid alone.

C.O.C.

Cadmium Orange (C.I. 77199)

K. N. Moorthy, Y. Venkataraman, and D. S. Datar

Research Ind. (New Delhi), 6 (1961) 45-46*Chem. Abs.*, 55 (24 July 1961) 14755

This pigment has been produced on a laboratory scale by dissolving Cd metal in a mixture of H_2SO_4 and HNO_3 , and precipitating with Na_2S . The product compared well with the imported pigment.

C.O.C.

Polycyclic Aromatic Hydrocarbons in Carbon Blacks

A. J. Lindsay and M. A. Phillips

Chem. and Ind., No. 30 (29 July 1961) 1171-1172

Samples of Carbon Black (C.I. Pigment Black 6 and 7) from an American source were found (by Soxhlet extraction) to contain only traces of polycyclic aromatic hydrocarbons, in contrast to some Carbon Blacks obtained as a by-product of butadiene manufacture, which may contain up to 13% aromatic hydrocarbons.

F.J.

Colour of Synthetic Ferric Oxide (C.I. 77491) and its Hydrate (C.I. 77492). III—Colours of Hematite and Maghemite

M. Kojima

Nippon Dojo Hiryogaku Zasshi, 30 (1959) 176-180*Chem. Abs.*, 55 (7 Aug 1961) 15231

X-ray diffraction patterns, electron-microscope photographs, and spectrophotometric observations of 12 synthetic and one natural hematite and two synthetic maghemites were used to calculate λ_d , P_d , and Y . λ_d of hematite is about 598 m μ , irrespective of particle size and degree of crystallisation, and that of maghemite about 590 m μ .

C.O.C.

Basic Lead Silicochromate: an Anticorrosive Pigment

R. P. Bates and R. L. Whitney

Off. Dig. Fed. Soc. Paint Technol., 33 (Jan 1961) 27-39*Chem. Abs.*, 55 (24 July 1961) 14933

Basic lead silicochromate pigment consists of 29-1% basic lead chromate (I), 25-5% tribasic lead silicate (II) and 45-4% SiO_2 (III), II and III being on the surface of a SiO_2 core. The pigment is dark orange, has d 4.1 and hiding power 390 ft² (at 98% contrast and 10 lb/gal loading). On carefully prepared steel panels this pigment gives considerably better resistance against corrosion than does Red Lead (C.I. Pigment Red 105) in either linseed oil or alkyd formulations, but is only slightly better than Red Lead on poor surfaces. Coatings containing the pigment showed no chalking, checking, cracking, or peeling after exposure for 2 years. The pigment has low tinting strength, so that strong colours readily mask its colour, whereas weak ones require addition of TiO_2 (C.I. Pigment White 6) to mask the colour of basic lead silicochromate. It is readily dispersed and can be used in both finish and prime coats.

C.O.C.

Colour and Constitution of the Isoalloxazine-Silver Complex

P. Bamberg and P. Hemmerich

Helv. chim. Acta, 44 (15 June 1961) 1001-1011

The strong bathochromic shift of the flavin spectrum occurring by interaction with Ag^+ can also be obtained with Cu^+ and Hg^{2+} . Two kinds of metal-isoalloxazine

interaction are found: a loose one with the free ligand and a very strong one with the ligand anion. 1:1-Complexes are built up preferentially and are trigonal chelates of the oxinate type whose colour is due to electron transfer from Ag^+ to flavoquinone, giving a Ag^{2+} -flavosemiquinone mesomeric state responsible for the specific chelating qualities. H.H.H.

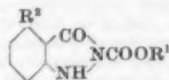
PATENTS

Colour Couplers

ICI

Compounds of formula

BP 875,470 (18 July 1958)



(R^1 = subst. or unsubst. hydrocarbon; R^2 = subst. or unsubst. hydrocarbon, COOR^3 or $\text{CON}(\text{R}^4)\text{R}^5$ (R^3 = subst. or unsubst. alk; R^4 and R^5 = H or subst. or unsubst. hydrocarbon)), e.g. carboxystyrene-*n*-butylcarbamyl-indazolone, are yellow colour couplers. C.O.C.

Colour-coupling Developers

DuP

BP 875,604 (U.S.A. 18 Nov 1958)

A silver halide emulsion layer containing as colour former a compound which reacts with tetracyanoethylene to form a dye is treated with a colour-coupling developer of formula



(A = arylene; n = 0 or 1; X = CN, COOR, CONH₂, CONHR, CONR₂, COR, NO₂, *p*-nitro-, or *p*-cyano-aryl; Y = CN, COOR, CONH₂, CONHR, CONR₂, COR, SO₂R, NO₂, F, Cl, Br, OR, ONO₂, OSO₂R or OCOR (R = Alk, cycloalkyl, Ar or aralkyl of 1-18 C)). Thus an emulsion containing *m*-dimethylaminophenol is exposed and then developed with a solution containing tetracyanoethane, NaHSO_3 , KBr, and Na_2CO_3 , rinsed, then bleached with a solution of K ferricyanide, boric acid and borax, washed, and fixed with hypo. This yields a magenta image. C.O.C.

***p*-Aminobenzene Diazo Compounds for Use in One-component Diazotype Material**

Chemische Fabrik van der Grinten

BP 875,307 (Holland 10 Nov 1958)

p-Aminobenzene diazo compounds derived from a tertiary amine having only one Ar attached direct to the tertiary N atom, a diazo group in the 4-position, an alkoxy, aralkoxy or aryloxy group in the 2-position, and H, Hal, CH₃ or alkoxy in the γ -position, have greater coupling activity than *p*-diazoethylbenzylamine. They are very suitable for use in the manufacture of one-component diazotype material to be developed by the semi-wet process. C.O.C.

Monoazo Pyrazolone Pigment Lakes

FBy

BP 874,220 (Germany 21 Feb 1959)

Diazotised 5-nitroanthranilic acid is coupled with 1-aryl-5-pyrazolones containing at least 1 SO₂H or COOH group, and the products are laked with salts of divalent metals to give yellows of good fastness to solvents. Thus the monoazo compound 5-nitroanthranilic acid \rightarrow 3-methyl-1-*p*-sulphophenyl-5-pyrazolone is heated with aq. BaCl_2 to give a yellow suitable for nitro-alkyd resin lacquers. E.S.

Monoazo Dyes and Pigments

FBy

BP 875,882 (Germany 2 Jan 1958)

Diazotised 5-nitro-2-aminobenzene-1-carboxylic acid amides (I) (in which the amide N is disubstituted) are coupled to two varieties of coupling component containing COOH and/or SO₂H groups. The azo dye may be converted by known methods into lakes of good fastness to light and solvents. They are suitable for colouring lacquers, plastics, textiles, and paper by conventional pigment dyeing and printing methods, and for the mass-coloration of man-made fibres. I are obtained by known methods, e.g. nitration of



and condensation with secondary amines. Thus the azo dye 5-nitro-2-aminobenzene-1-carboxylic acid dimethylamide \rightarrow 1-(4'-sulphophenyl)-3-methyl-pyrazolone-(5) is heated for 2 h at 90°C in acetic acid in presence of BaCl_2 . The resulting pigment, ground in a hopper mill with a nitro-alkyd resin lacquer, yields a yellow lacquer of very good fastness to light and top spraying. A.T.P.

Azo Pigments of Improved Physical Form

General Aniline

USP 2,938,897 (28 Dec 1955)

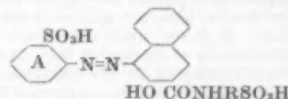
By coupling in presence of suitable anionic surface-active agents (25-175 g per g mol of pigment), azo pigments can be produced in a finely divided form. On boiling, flocculation occurs, so that the product can be readily filtered off, but it can readily be redispersed by a mild milling. E.S.

Alkaline-earth-metal Lakes of Benzeneazo-naphthalene Dyes

FH

BP 875,871 (Germany 23 Aug 1957)

Red pigments of outstanding fastness to light and migration are the Ba, Ca, and Sr lakes of the azo dyes



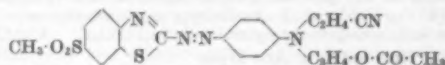
(A has no other SO₂H but may contain Hal, Alk or alkoxy; R = benzene or naphthalene nucleus with SO₂H *para* to NH). They are suitable for colouring high-mol.-wt. synthetic resins, e.g. polyvinyl chloride, and, because of their great resistance to heat, stoving enamels. They have better fastness to solvents and bleaching than the pigments of BP 751,447 (J.S.D.C., 72 (1956) 451). Thus, the dye 1-amino-4-chloro-5-methylbenzene-2-sulphonic acid \rightarrow 1-(2',3'-hydroxynaphthylamino)benzene-4-sulphonic acid is stirred at 90-95°C in aq. BaCl_2 to give a yellowish red lake of excellent fastness to solvents, migration, and light. A.T.P.

Disperse Monoazo Dyes from 2-Aminobenzothiazoles

Eastman Kodak Co.

BP 876,090 (Switzerland 11 Sept 1956)

Monoazo disperse dyes of the type 2-amino-6-alkyl(or allyl)sulphonylbenzothiazole \rightarrow *N*-hydroxyalkylaniline are *O*-acylated to give generally improved brightness of hue. Thus the monoazo compound 2-amino-6-methylsulphonylbenzothiazole \rightarrow *N*- β -cyanoethyl-*N*- β -hydroxyethyl-aniline treated with acetic anhydride gives



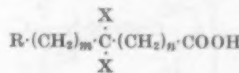
which dyes cellulose acetate bright scarlet. E.S.

Azo Dyes from Bis-phenolic Compounds

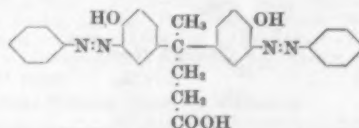
Quaker Oat Co.

USP 2,945,848 (20 Aug 1957)

Mono-, di-, and poly-azo dyes are prepared by coupling suitable diazo compounds with compounds



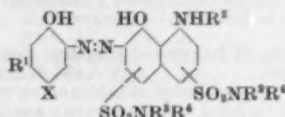
(R = H or COOH; m = integer from 0 to 15; n = integer from 0 to 4; X = residue of phenol or naphthol having at least one reactive position on the ring) prepared by condensing 2 mol. of a suitable phenol or naphthol with 1 mol. of a keto acid. Thus phenol and laevulinic acid condensed at 50°C in 37% aq. HCl by the method of BP 768,206 give 4,4-bis(*p*-hydroxyphenyl)valeric acid. Coupling with 2 mol. of diazobenzene gives the diazo compound



which dyes wool golden yellow. E.S.

Blue Metal-complex Monoazo Dyes for Wool, Nylon, etc.

General Aniline USP 2,938,896 (28 Dec 1955)
The metal (especially Cr) complexes of monoazo compounds



(R¹ = NO₂, NH₂, or NHAcyl; R² = H or Acyl; R³, R⁴ = H, Alk, hydroxyalkyl, or together form part of a heterocycle; X = H, Cl, or Br) dye wool, nylon, etc. blue from neutral or weakly acid baths. They are obtained by coupling diazotised 2-amino-5-nitro-4-X-phenols with suitable disulphonamides of 1-acetylamino-8-naphthols, followed if desired by reduction and acylation of the nitro group and hydrolysis of the acyl group R². Thus diazotised 2-amino-4-chloro-5-nitrophenol is coupled with a solution in pyridine of 1-acetylamino-8-naphthol-3,6-bis(sulphon-dimethylamide). Heating with chromium formate in formamide followed by reduction of the nitro group with Na₂S gives the Cr-complex of the aminoazo compound, which dyes wool greenish blue from a neutral bath. E.S.

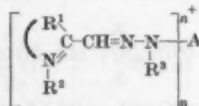
Azo Disperse Dyes for Protein Fibres

ICI BP 875,106 (8 May 1959)
Azo disperse dyes free from sulphonie or carboxylic groups but containing at least one -CO-Y-A-Z group (Z = Hal or oxiranyl; A = subst. or unsubst. alkylene; Y = a direct link or NH, >NB or >N-A-Z (B = subst. or unsubst. Alk or Ar)), each such group being attached to a C atom of the dye nucleus, have good affinity for protein fibres. Applied either by dyeing or printing they yield dyeings of excellent fastness to wet processing. Thus wool dyed with a dispersion of the brown 2-bromo-4-chloroacetyl-4'-NN-bis(β-hydroxyethyl)aminoazo-benzene with NaH₂PO₄ and disodium methylenedinitrophenylsulphonate as assistants for 1 h at the boil, has excellent fastness to washing and milling.

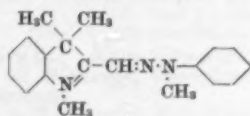
BP 875,451 (8 May 1959)
Disperse dyes containing at least one -Y-A-Z group (Y = O or S; A = subst. or unsubst. alkylene; Z = Hal or oxiranyl group) but no sulphonie or carboxylic acid group are readily applied to protein textiles from aqueous dispersion. Thus wool is dyed a level orange of good fastness to wet processing in an aqueous bath containing 1-(4'-(γ-chloro-β-hydroxypropoxy)phenylazo)-2-naphthol, a dispersing agent, and Na₂HPO₄. C.O.C.

Basic Heterocyclic Azo Dyes

FBY BP 875,995 (Germany 30 Aug 1957)
The hydrazo forms of basic azo and azomethine dyes derived from heterocyclic compounds having a reactive methylene group or a methylene-α-aldehyde group are treated with quaternising agents, e.g. dimethyl sulphate, to give basic dyes



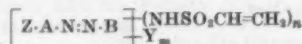
(R¹ = residue of 5- or 6-membered ring; R² = Alk, Ar, or aralkyl; R³ = Alk, aralkyl, or cycloalkyl; A = mono- or di-valent radical of an aromatic or heterocyclic amine; n = 1 or 2). Thus the hydrazo dye from diazobenzene and 1,3,3-trimethyl-2-methylene indoline is treated with aq. Na₂CO₃, recrystallised, and then dissolved in toluene and treated with dimethyl sulphate to give



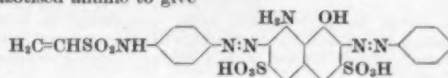
which dyes cellulose acetate and polyacrylonitrile fibres yellow. E.S.

Water-soluble Reactive Azo Dyes Containing Ethene Sulphonie Acid Amide Groups

BASF BP 875,946 (Germany 10 Oct 1958)
The above dye natural and synthetic-polymer fibres, especially cellulose, faster to washing than the vinyl sulphone types of BP 712,037; BP 776,265; and BP 779,781 (J.S.D.C., 70 (1954) 465; 73 (1957) 476, 524), and in many cases have better absorptivity. They are



(A = subst. or unsubst. benzene nucleus; Z = H or subst. or unsubst. benzene, linked to A via CH₂, CO, NH, SO₂, SO₂NH or S; B = residue of phenolic, naphtholic, enolic, or heterocyclic coupling component; Y = water-solubilising group; n = 1-2; m = 1-4) and are obtained by acylating (to introduce ethene sulphonie acid radical) an azo dye containing < 1 acylatable NH₂ group and < 1 water-solubilising group, or by acylating either the diazo or coupling component before dye formation. Thus diazotised ethene sulphonie acid-(4-aminophenyl)amide is acid-coupled to 1-amino-8-hydroxynaphthalene-3,6-disulphonie acid, and the monoazo dye further coupled with diazotised aniline to give

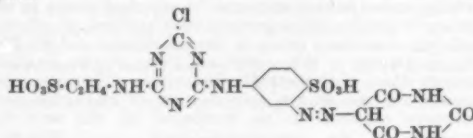


which dyes cotton a fast blue.

A.T.P.

Monoazo Reactive Dyes for Cellulose

Ciba BP 874,544 (Switzerland 14 Sept 1956)
m- or p-Aminomonazo compounds H₂N-R¹-N=N-R² (R¹ = benzene residue containing an SO₃H or COOH group; R² = residue of a 6-membered heterocyclic coupling component, especially barbituric acid) are condensed with 1 mol. of cyanuric chloride, and a second Cl in the cyanuric ring is then replaced by condensation with an aliphatic or aromatic amine containing a COOH and/or an SO₃H group, to give yellow reactive dyes. Or these reactions may be carried out in a different order to give the same products. Thus 1 mol. of cyanuric chloride is condensed first with 1 mol. of aminocethanesulphonie acid and then with 1 mol. of m-phenylenediamine-4-sulphonie acid. Diazotising and coupling with barbituric acid then gives

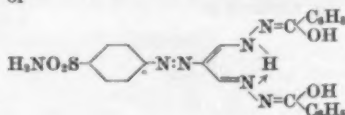


which dyes cotton greenish yellow when fixed by steaming in presence of NaOH.

BP 874,545 (Switzerland 25 Oct 1957)
Similar dyes are obtained if the second Cl atom of the cyanuric ring is condensed with dehydrothio-p-toluidine-monosulphonie acid instead of with an aliphatic or homocyclic amine sulphonie or carboxylic acid as described above. E.S.

Metalliferous Dyes Derived from Arylazomalonaldehydes

BASF BP 875,786 (Germany 11 July 1957)
Arylazomalonaldehydes (1) (see GP 947,468) are condensed with carboxylic acid hydrazides (2) and the resulting dihydrazone is metallised to give dyes for natural and synthetic-polymer fibres (especially polyamides), lacquers, and plastics. Thus, 4-aminosulphonyl-phenylazomalonaldehyde (10) is heated for 20 min at 60-70°C in dimethylformamide (300) with benzhydrazide (11). Crystalline Ni acetate (10) in water (50) is added and, after stirring for 2-4 h at 80-90°C and cooling, the Ni complex of

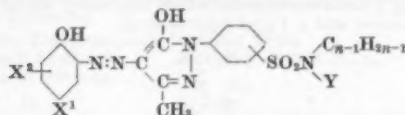


is obtained; it dyes polyamide fibres brilliant red. 102
Dyes are described. A.T.P.

Chromiferous Benzene Monoazo Pyrazolone Dyes

Ciba BP 875,839 (Switzerland 30 Dec 1957)

These are, especially, chromiferous (1 : 2) complexes of

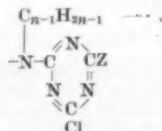


($n = 1-3$; $X^1 = \text{Cl}$ or Alk ; $X^2 = \text{NO}_2$ or Cl ; $\text{Y} = \text{benzene residue which may be hydrogenated}$) prepared by standard methods. They colour lacquers and spinning compositions act as transparent dyes for lacquers, colour natural and artificial resins, cellulose derivatives (celluloid) or animal substances (casein, gelatin), are suitable for dyeing synthetic-polymer fibres, and, when sufficiently water-soluble, wool, silk, and leather. They have purer colours than the dyes of BP 719,274 and BP 793,712 (J.S.D.C., 74 (1958) 560). Thus, 6-nitro-4-chloro-2-aminophenol is diazotised and coupled to 1-phenyl-3-methyl-5-pyrazolone-3'-sulphonic acid-N-phenylamide, and the moist press cake refluxed in aq. NaOH with Na chromosalicylate to give a metallised product which dyes wool and polyamides bluish reds of good fastness. A.T.P.

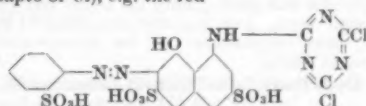
**Coloured Synthetic Resin and Dyes therefor—
Moulding Powders and Pigments**

Ciba BP 874,369 (Switzerland 23 Sept 1957)

Dyes containing at least one acid group conferring solubility in water and at least one group containing at least one labile halogen atom, particularly dyes containing a group of formula



($n = 1-4$; Z = subst. or unsubst. amino, subst. hydroxy or mercapto or Cl), e.g. the red



can be added to aminoplast precondensates before these are cured. The products are suitable for moulding powders or as pigments.

C.O.C.

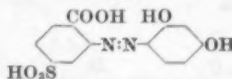
Reactive Azo-Phthalocyanine Dyes for Cellulose

CFM *BP 874,355 (Germany 7 Aug 1958)*

The title dyes contain a phthalocyanine residue to which are attached 1-4 groups $-SO_2R^1(X)R^2N:N:R^3N(X)Y$ ($X = H, Alk, Ar, etc.$; $R^1 =$ residue of azo coupling component; $R^2 =$ arylene; $Y =$ residue of halogeno fatty acid or mono- or di-halogenotriazine). Thus copper phthalocyanine is stirred in chlorosulphonic acid at $140^\circ C$, thionyl chloride is added at $80-90^\circ C$, and the sulphochloride so formed is condensed with 1-m-aminophenyl-5-pyrazolone-3-carboxylic acid. Coupling with diazotised 1-(β -chloropropionylamino)-3-aminobenzene-4-sulphonic acid then gives the yellowish green reactive dyes. E.S.

Metal-complex Mono- and Dis-azo Dyes for Leather
FH BP 875,990 (Germany 26 Jan 1957)

The title dyes are Co, Cu, Cr, or Ni complexes of dyes formed by coupling 1 or 2 mol. of diazotised sulphoanthranilic acids (which may contain NO_2 or CH_3 groups as substituents in the benzene ring) with 1 mol. of resorcinol or its carboxylic acids. Thus 1 mol. of diazotised 4-sulphoanthranilic acid coupled with resorcinol gives

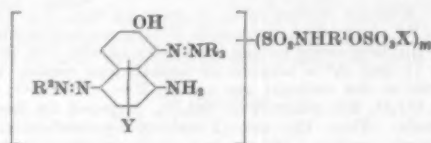


Heating with aq. chrome alum gives a reddish brown leather dye. E.S.

Black Disazo Dyes Containing Acid Sulphate Ester Groups and Metallised Complexes thereof

FBy BP 875,888 (Germany 31 Jan 1958)

Disazo dyes which may be metallised for hydroxy- or amide-containing fibres are

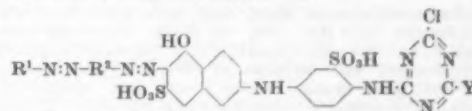


(Y = H or SO₃H; R² = benzene residue; R³ = benzene residue carrying a metallisable group *ortho* to azo; R⁴ = ethylene radical; X = H or alkali-metal cation; m = integer). They may be obtained by acid coupling diazotised aniline derivative to 1-amino-7-hydroxynaphthalene (or a monosulphonic acid thereof) and further coupling (alkaline) the monoazo dye with an aniline deriv. containing a metallisable group (or precursor of), so that < 1 of the diazo components contain < 1 -SO₃NHR²OSO₃X group, and with final metallisation (Cu, Co, or Cr) in substance or on the fibre. Other preparations are described. They are reactive dyes for cotton, whereas on wool and silk they are applied by known methods to give dyeings of outstanding fastness to washing and very good fastness to light. Thus, 1-amino-2-nitrobenzene-4-sulphonylamino ethylhydrogen sulphate is diazotised and acid coupled to 1-amino-7-hydroxynaphthalene, and the resulting monoazo dye alkaline-coupled with diazotised 6-chloro-2-amino-1-hydroxybenzene-4-sulphonylaminoethyl hydrogen sulphate. The product dyes cotton black of excellent fastness when applied from a bath containing NaOH-urea and aftertreated in acetic acid-CuSO₄ solution. A.T.P.

Violet and Blue Disazo Reactive Dyes for Cellulose

Ciba BP 876,092 (Switzerland 14 Sept 1956)

The title dyes are of the type

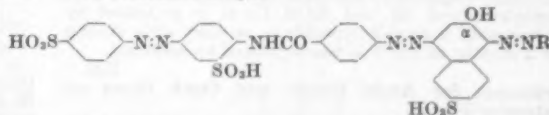


RU = benzene, naphthalene, or pyrazolone residue having an SO₃H group; R² = benzene residue with the azo groups in the *m*- or *p*-position to each other; Y = NH₂, NHalk, or NHA₂. Thus 4-aminoazobenzene-3,4'-disulphonic acid is diazotised and alkali-coupled with *p*-amino-*m*-sulphophenyl-J acid. Condensation with 1 mol. of cyanuric chloride, followed by treatment with ammonia to replace a second Cl of the cyanuric ring by NH₂, gives the violet reactive dye. E.S.

Cupriferous Trisazo Dyes for Cellulose

Capricious Itrazo Dyes for Cellulose
Fran BP 875-654 (France 27 Mar 1958)

Cu complexes of dyes



(R = residue of acylaminohydroxynaphthalene sulphonic acid coupled *ortho* to OH) may be obtained by dealkylating metallisation (with divalent Cu compounds) of the products with O-Alk at α . Where R = 6-acylamino-1-naphthol-3-sulphonic acid residue (coupled at 2) any excess of Cu may also be fixed to give 2:1 complexes, readily converted into 1:1 complexes by heating at 80°C with tetrasodium ethylenediamine tetra-acetate either before or during dyeing. They dye vegetable fibres fast to light and washing. Thus the 1:1 complex of the dye where R = 7-benzamido-1-naphthol-3-sulphonic acid is a fast bluish green. A.T.P.

Cupriferous Trisazo Dyes for Cellulose

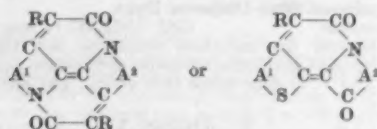
FBv BP 875.672 (Germany 10 Sept 1958)

Green dyes of excellent fastness on natural and regenerated cellulose are

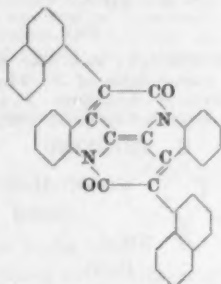
Dyes for Mass Coloration of Organic Polymers of High Melting Point

Ciba BP 875,039 (Switzerland 24 Dec 1957)

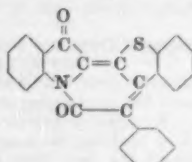
Dyes of formula



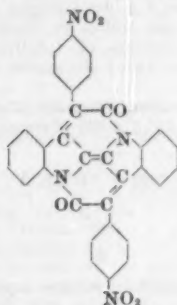
(A¹ and A² = arylene containing no water-solubilising group; R = Ar containing no water-solubilising group, e.g. the scarlet



made according to BP 8421:1912, the orange



made according to GP 263,470, and the violet



made according to BP 8421:1912, are excellent dyes for mass-colouring nylon, polyesters, etc., which are to be melt-spun. C.O.C.

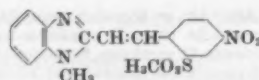
Benzimidazolydene Compounds as Fluorescent Brightening Agents or Intermediates therefor

Gy BP 874,209 (Switzerland 21 Dec 1956)

Condensing a compound of formula $X-CH_2-Y$ (X = an electrophilic group; Y = H, an inert organic group, or an electrophilic group) and having an activated methyl or methylene group with benzimidazole-2-aldehydes containing no substituents in the benzene ring, the reaction being carried out by splitting off water in presence of a catalyst, yields compounds, some of which are fluorescent brightening agents, e.g.



which is substantive to cellulose, nylon, and polyester fibres, and the deep yellow

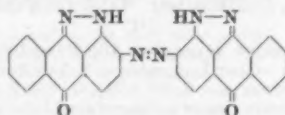


which on acylation becomes only slightly yellow and shows a vivid bluish violet fluorescence. C.O.C.

Azopyrazoleanthrones—Dark Red and Violet Vat Dyes and Pigments

American Cyanamid Co. USP 2,962,494 (12 Oct 1959)

When 1-chloro-2-nitroanthraquinone is treated with a large excess (10:1) of hydrazine, the dye



which dyes cotton dark red from a deep blue vat, is obtained. When treated with metallic salts in dimethylformamide solution, it readily yields chelates which are of use as pigments. Replacing H in the above formula by Alk or aralkyl yields vat dyes which can also be used for pigmenting resins and hydrocarbons. Thus, replacement of H by CH_3 yields a dye which dyes cotton violet from a green vat and which, incorporated in plasticised polyvinylchloride, colours it brown. C.O.C.

Colourless Carbon Papers and Heterocyclic Compounds therefor

Allied Chemical Corp.

BP 875,764 (U.S.A. 12 May 1958)

N-bis-(p-dialkylaminoaryl)methyl derivatives of unsaturated heterocyclic nitrogen compounds containing no carbonyl groups adjacent to the ring N and > 4 atoms in the ring, the alkyl groups in the dialkylamino radicals each being of 1-5 C and the aryl groups mononuclear, when dissolved in a non-polar solvent yield deep colorations when brought into contact with an acidic electron acceptor. Thus the pale yellow bis-(p-dimethylamino-phenyl)benzotriazylmethane, dissolved in toluene and formed into a coating of attapulgite, produces a deep blue coloration within a few seconds. Such compounds are used to produce colourless carbon paper either by impregnating paper with them and using them on a copy paper coated with attapulgite or other acidic electron acceptor, or by impregnating paper with discrete particles of each component dispersed in a solid insulating material so that they come into contact with each other only when pressure is applied. C.O.C.

Phthalocyanine Pigments

DuP

USP 2,964,532 (17 Apr 1957)

A layer 0.25-0.5 in. thick of a mixture of phthalocyanine-forming ingredients is passed at reaction temperature between and in contact with the inner surface of a cylinder and the surface of a screw rotating within the cylinder, the thickness of the layer being controlled by the size of the screw's shaft. This enables continuous production of the pigment to be readily controlled and avoids use of a solvent. C.O.C.

Acid Pasting of Phthalocyanine Pigments

Pittsburgh Chemical Co.

USP 2,964,531 (6 Nov 1956)

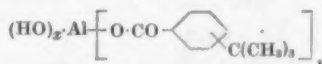
Acid pasting or milling at 45-100°C in presence of an arylene dicarboxylic acid, an alkane dicarboxylic acid, cycloalkene dicarboxylic acid, or an anhydride of one of these acids, can be carried out at 45-100°C for 1-8 h without any change in hue occurring. This yields a very finely powdered product much freer from grit than the pigment obtained by normal acid pasting and of much stronger masstone, and cleaner and brighter colour. C.O.C.

Non-flocculating Phthalocyanine Pigment

Standard Ultramarine & Color Co.

USP 2,965,511 (24 June 1955)

Incorporation of a compound of formula



(y = 1, 2, 3; x = 3 - y) with a phthalocyanine pigment yields a product which does not flocculate and separate

out when dispersed in an organic vehicle. It is preferable that the stabiliser, e.g. Al tri-*p*-*t*-butylbenzoate be precipitated or formed on the pigment, though dry mixing is effective, and that the pigment shall form 40–60% of the resulting product. C.O.C.

β-Carotene (C.I. 75130)

Grain Processing Corp. USP 2,959,522 (12 Jan 1959)
β-Carotene is obtained by cultivating a carotene-producing strain of *Choanephora* in a nutrient containing one or more terpenoid substances, e.g. cedarwood oil.

USP 2,959,521 (12 Jan 1959)

Cultivation is carried out in a nutrient containing one or more of the following: 2,6-di-*t*-butyl-4-methylphenol, propenylmethylguaiacol, α-tocopherol, NN'-dibutyl-*p*-phenylene diamine, 6-ethoxy-1,2-dihydro-2,3,4-trimethyl-quinoline, ascorbic acid, hydroquinone, α-naphthol, lecithin, propyl gallate, ascorbyl palmitate, ethylhydrocatechate, 3,4-dihydrocoumarin, and thiopropionic acid. The yield is increased if an ionone and/or a terpinoid is also present. C.O.C.

Precipitated Calcium Carbonate (C.I. 77220)

Wyandotte Chemicals Corp.

USP 2,964,382 (4 Apr 1958)

CaCO₃ is obtained in non-composite state by contacting Ca ions with CO₃ ions in aqueous medium maintained at pH < 8.5 while subjecting the solution and the precipitated pigment to anticompositing forces of intensity and magnitude sufficient to keep all of the crystals completely segregated. Such anti-compositing forces are obtained, e.g., by use of the apparatus described in USP 2,619,330. C.O.C.

Whiting (C.I. Pigment White 18)

Aluminum Laboratories

USP 2,962,350 (3 Dec 1957)

Whiting of high purity is obtained by first slurring an impure MgO containing material in an aqueous solution of MgCl₂ through which CO₂ is bubbled (the "carbonator-chloride" stage). This produces a solution of magnesium bicarbonate and magnesium chloride with a residue of impure whiting. This solution is fed in a "whiting precipitator" stage into aqueous CaCl₂ (10% excess of CaCl₂). This yields precipitated CaCO₃ and a solution of MgCl₂. One half of this solution is recycled as feed to the carbonator-chloride stage. The second half of the solution is fed to a "magnesium hydroxide precipitator" stage where it is mixed with impure lime. This produces magnesium hydroxide and a solution of CaCl₂ which forms the feed for the whiting precipitator stage. C.O.C.

Carbon Black (C.I. Pigment Black 6 and 7) Pellets for News Ink

J. M. Huber Co.

USP 2,960,413 (22 Aug 1957)

A fluffy Carbon Black of alkaline reaction when dispersed in water is treated with a material which renders smooth-flowing a buttery dispersion of the pigment in mineral oil and then pelleted to an apparent density of 18 lb/ft³. This yields strong pellets which are readily dispersed in ink oil. Thus fluffy Channel Black of apparent density 4 lb/ft³ of pH (in water) 8.2 was sprayed with 8% of its weight of the tar-like residue obtained by the propane dewaxing of an asphalt-base petroleum, and then formed into pellets of bulk density 18 lb/ft³. It readily disperses in medium-viscosity ink oil to yield an acceptable ink (325-mesh-screen residue of only 0.003%) whereas a similar pigment but of pH 4.6 in water, treated similarly, was not usable (screen residue 3.1%). C.O.C.

Controlling the Characteristics of Carbon Black (C.I. Pigment Black 6 and 7) during Hot Pelletizing

Phillips Petroleum Co.

USP 2,964,392 (12 Nov 1957)

A method of improving the pelletizing of the pigment with simultaneous control of its modulus and photometer (light transmission) of a suspension of 2 g pigment in 50 ml CCl₄ compared with that of the pure solvent). C.O.C.

Carbon Black (C.I. Pigment Black 6 and 7) Slurries

Phillips Petroleum Co.

USP 2,965,510 (6 June 1958)

Aqueous slurries are produced with little or no foaming by mixing the pigment with water containing a rosin acid soap, an alkali-metal or ammonium hydroxide, and an antifoam agent, the proportions of the components being pigment (100), rosin and soap (5), alkali (< 0.2), water (to give solids content of 8–20%) and antifoam agents (0.005–0.20). The antifoam agent consists of an aliphatic alcohol of 14–25 C (55–72% by wt.), aliphatic acid of 14–

25 C (3–25) and an alkali-metal or amine soap of an aliphatic acid of 14–25 C (5–42). The slurries do not foam when used, e.g. when added to latex. C.O.C.

Nacreous Lead Pyrophosphate

Sherwin-Williams Co.

USP 2,959,464 (3 Aug 1953)

Lamellar lead acid phosphate, prepared preferably by the process of USP 2,703,747 (U.S.D.C., 72 (1956) 117) is rendered stable up to 700°C and fast to ultraviolet radiation by heating the crystals of particle size 20–60 μ × 30–120 μ at 225–700°C until they have constant weight. C.O.C.

Removing Chromium from Titanium Dioxide

Horizons

USP 2,960,387 (14 Mar 1958)

The hydrated or calcined rutile obtained by the process of USP 2,914,381 (U.S.D.C., 76 (1960) 604) is leached in caustic soda containing a small amount of a peroxide. This yields a product containing < 0.0015% Cr and free from pyrites, SiO₂, aluminates, and alkaline-earth compounds, and useful for processes in which heavy-grade rutiles are required, e.g. for white or coloured porcelain enamels. C.O.C.

Removing Grit from Carbon Black (C.I. Pigment Black 6 and 7) (I p. 562)

Azoic Dyeings using Chloronaphthylamines (VIII p. 581)

Studies on the Dyeing of Acrylonitrile Fibre. IX—Purification and Estimation of Acid Dyes (VIII p. 582)

Dyeing Cellulose Triacetate with 1:2 Metal-complex Unsulphonated Monoazo and Monoazomethine Dyes (VIII p. 583)

V—PAINTS; ENAMELS; INKS

Production and Control of High Dry Hiding

F. B. Stieg and R. I. Ensminger

Off. Dig. Fed. Soc. Paint Technol.,

33 (July 1961) 792–806

Relative ability of a white pigment to produce dry hiding (DH) may be expressed by

$$HP = 4430 R \left(1 - \sqrt[3]{\frac{PVC}{0.74}} \right)$$

where HP = ft²/lb at 0.98 contrast ratio, *R* = Fresnel reflectance in air, and PVC = pigment vol. concn. In formulations for high DH the PVC term in the equation is the critical PVC of the pigment itself. Of two pigments having similar refractive indices, the one having the lower critical PVC gives the higher degree of DH. Coarse extenders contribute practically nothing to DH power, irrespective of film porosity. Paint-film porosity can be expressed in terms of its porosity index

$$\text{Porosity index} = 1 - \left(\frac{CPVC}{1 - CPVC} \right) \left(\frac{1 - PVC}{PVC} \right)$$

where CPVC = critical pigment vol. concn. Porosity index and DH power at different PVC levels are linearly related for any given pigmentation. Rate of development of DH for a given sacrifice in film integrity differs considerably with pigmentation. High percentage of prime pigment and small particle size of extender favour rapid increase in DH with increased porosity. By a suggested procedure the DH power of a given pigmentation over a wide range of PVC may be predicted by means of only two hiding-power determinations. It is recommended that the porosity index factor should be used in the identification of acceptable porosity levels for various formulations. J.W.D.

Red Iron Oxides for Rust Preventive Primers

W. G. Wade

Deutsche Farben-Zeitschrift, 15 (July 1961) 287–290

The protection against corrosion afforded by iron oxide pigments in priming paints arises mainly from a barrier effect and from the absorption of deleterious ultraviolet radiation. Selection of the iron oxide pigment is not governed by the iron oxide content but by the amount of water-soluble salts present. Selected natural oxides contain < 0.4% salts (mainly calcium sulphate), whereas synthetic oxides can contain corrosive chlorides and sulphates. The best natural oxides for primers usually contain 80–87% Fe₂O₃ with neutral clay and silica, and synthetic oxides contain 97% Fe₂O₃. Oxides of low oil

absorption value (natural: 12–14, synthetic: 17–25) give better barrier protection, and micronising, which does not change oil absorption, favours the development of a consolidated pigment layer by reducing coarse particles. Highly ionised salts (ferrous and sodium sulphates and most chlorides) are deleterious, but calcium sulphate is less active and can be tolerated in small amounts. The alkali and silicate salts of iron are mildly protective, but excessive alkali can attack the resinous component of the primer and react with the driers. Oxides which give acidic aqueous extracts are also to be avoided. BS 272 and 305 specify iron oxides but are inadequate to define optimum qualities for primers. A reliable and rapid test procedure for assessing the corrosion protection of primers is available in DEF 1053. Natural oxides may settle from the paint on storage, but micronising overcomes this defect and reduces the tendency to pigment "floating" on application. These defects are rare with synthetic oxides. Drier adsorption occurs with all oxides, but adding zinc naphthenate first protects against the adsorption of cobalt naphthenate added later.

E.C.

PATENTS

Accelerators for Driers

R. T. Vanderbilt Co. USP 2,961,331 (3 Oct 1958)

2-(2-Pyridyl)-benzimidazole, 1-(2-pyridylazo)-2-naphthol, 2-picolyamine, 2,2'-dipyridylamine, 2-pyridine-methaldoxime, 2-pyridinealdoxime, 2-(2-pyridyl)-benzoxazole, 2-pyridinaldazine and bis-(2-pyridinal)-ethylene-diamine accelerate the drying action of metal carboxylates. Their use also yields compositions of good drying time stability after ageing. Films obtained by use of aged compositions containing these accelerators give a good solubility test.

C.O.C.

Non-toxic Lead Paints

Associated Electrical Industries

BP 873,917 (23 July 1958)

Use of a chelated compound of lead, e.g. Detarate (F. W. Berk & Co.), a lead disodium compound of ethylenediaminetetra-acetic acid, in a normal paint vehicle, together with a drier and a non-toxic pigment or dye, yields a paint which is non-toxic.

C.O.C.

Metallic, Reflective Coating on Glass Beads for Incorporation in Coating Compositions

Minnesota Mining & Manufacturing Co.

USP 2,963,378 (25 Apr 1955)

Glass microspheres of diameter 10–50 μ are coated with metal over one half of their surface. They are readily mixed with varnish and lacquer, and if they do settle out are easily redispersed by stirring. Varnishes and lacquers containing such microspheres can be applied by spraying, painting, printing, or through stencils, to yield coatings which are highly reflective of light falling on them. Textiles treated with a dilute spray of such a coating composition show little alteration in their daylight appearance, nor is their handle appreciably affected, but they become highly visible when illuminated at night, e.g. by a car's headlights.

C.O.C.

Multicolour Coating Compositions

Airkem

USP 2,964,417 (14 Sept 1959)

A film-former is dispersed in a different type of film-former to produce a composition which yields continuous films in which the disperse phase is enveloped by and distributed in random fashion in the continuous phase. The disperse phase may consist of several portions of the same film-former, each containing a differently coloured pigment from the others, these portions being dispersed in the second type of film-former in succession to each other.

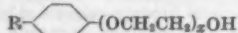
C.O.C.

Tinting Paste Vehicle

W. A. Cleary Corp.

USP 2,963,380 (17 Dec 1957)

A tinting paste vehicle which yields pastes which are (a) compatible with conventional paint vehicles, (b) readily dispersed, and (c) develop full colour in organic and aqueous solvent paint systems, comprises linseed oil and a surfactant of formula



(R = octyl or nonyl; x = 3–10), e.g. a mixture of linseed oil (80) and a t-octyl phenoxy polyethyleneglycol (20).

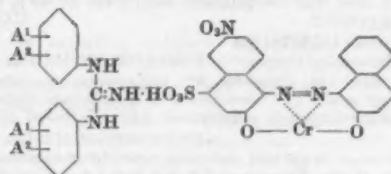
C.O.C.

Inks for Ball-point Pens

Allied Chemical Corp.

USP 2,966,419 (25 Nov 1957)

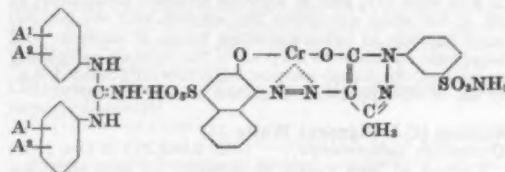
Compounds of formula



(A¹ and A² = H, CH₃, C₂H₅, C₃H₇, or iso-C₃H₇), e.g. the di-(o-ethylphenyl) guanidine salt of the free acid of C.I. 15711, when dissolved in a solvent of formula R¹O(R²O)_nH (R¹ = alkylene of 2–10 C; R² = H and Alk of 1–8 C; n = 1–5; total C = 2–10), e.g. octylene glycol, yield a well-defined black writing of good resistance to smearing, feathering, offset, and bleeding, and of excellent fastness to light.

USP 2,966,417 (3 Jan 1958)

Red inks of the same properties are obtained by using as the colorant a compound of formula



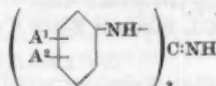
e.g. the diphenyl guanidine salt of the free acid of C.I. Acid Red 191.

USP 2,966,418 (10 Jan 1958)

The salts of diphenyl guanidine with the free acid of C.I. 16255 are brilliant scarlets of extremely high solubility in organic solvents but have only moderate fastness to light and to water. They can be used as the sole colorant in copying inks and for shading permanent inks.

USP 2,966,680 (25 Nov 1957)

The salts of the free acids of C.I. 42640 and C.I. 42735 with diaryl guanidines of formula



used as colorants in inks of the above types yield purple, royal blue, and violet of great strength and brilliance and of fastness to light and to water at least equal to that of ordinary inks. Used together with a copper phthalocyanine colorant they yield inks which combine depth of colour and brilliance with a fastness to light and to water superior to that of ordinary permanent inks.

C.O.C.

Metal Marking Ink

G. L. Jones

USP 2,962,398 (26 June 1959)

Zirconium oxychloride (20 g) and copper chloride (100) are pasted in water (700 ml), hydrochloric acid (300 ml) is added, and the mixture stirred until a clear solution is obtained. This gives an ink which, applied lightly by a suitable stamp to metal and neutralised after a few seconds by treatment with ammonia or a soluble cutting oil or dusting with a weak alkali, yields a permanent black mark on aluminium and an orange red mark on ferrous alloys.

C.O.C.

Coating Compositions for Metal and Paper

Dow Chemical Co.

BP 873,876 (12 Mar 1959)

An air-drying coating composition consists of a pigment dispersed in an aqueous latex of an interpolymer of an ethylenically unsaturated acid (2–10% by wt.), butadiene (30–50), and a 10–100:90–0 alkyl aromatic monomer-acrylonitrile mixture (48–68).

C.O.C.

Polymethylene Polyphenols (III p. 563)

Elastic Coating and Impregnating Compositions Compatible with Daylight Fluorescent Pigments (X p. 587)
Polyesters for Surface Coatings (XIII p. 591)

VI—FIBRES; YARNS; FABRICS

Selection of Fabrics for Filtration Processes

S. Ehlers *Ind. Eng. Chem.*, 53 (July 1961) 552-556

Problems associated with the correct choice of textile fabric to be used in a filtration process are enumerated. These include the character of the fluid and solid to be separated, the nature and build-up of the solid cake, the quantity of material to be processed and the filter fabric construction. Factors which enable a correct selection, with reference to the type of fibre and cloth structure, are given. These consist of chemical and physical stability of different fibre types, the influence of continuous-filament and staple yarn, fabric geometry and weave construction on the retention, production, moisture-content, and dischargeability of the solid, and max. life and min. binding of the fabrics used.

F.J.

A Quantitative Theory of the Loss in Strength of Regenerated Cellulose Filaments on Hydrolysis by Acid

R. J. E. Cumberbirch and C. Mack

J. Textile Inst., 52 (Aug 1961) T382-T389

A previously suggested model structure for regenerated cellulose is used to account quantitatively for the rate of loss in strength of cellulose filaments on hydrolysis by acid. Theoretical and observed values of loss in strength are in good agreement.

W.R.M.

States of Aggregation of High-molecular Compounds. VIII—Effect of Temperature on Ramie Fibres and Cellulose Hydrate Films

R. I. Fel'dman

Kolloid. zhur., 23 (No. 3) (May-June 1961) 315-321

Dilatometric measurements have been used to study the thermal expansion of ramie fibres and cellulose hydrate films. The results obtained indicate a relatively strong fixed structure in ramie fibres, where the changes produced by heating to 150-180°C are reversible. Comparison of shrinkage and hysteresis loops of ramie fibres with those of cellulose hydrate films shows that the structure of the latter is looser, but the changes produced by thermal treatment are also reversible. In the case of proteins and anisotropic synthetic polymers, e.g. polyvinyl chloride, polystyrene, etc., these changes are irreversible, indicating the higher lability of their structures.

I.G.

Apparent Increase in the Cystine Content of Heat-treated Wool

J. Barut and M. Leveau

Bull. Inst. Text. France, (94) (May-June 1961) 75-80

Heating wool at 105°C in air for 15 min produces a 3% increase in the apparent cystine content (colorimetric-phosphotungstic acid) without change in the cysteine or total sulphur contents. On further heating the apparent cystine content falls again. If the wool is heated under nitrogen, the increase is greater (5%) and there is no fall on subsequent heating. The apparent increase in cystine content is accompanied by a fall in the tyrosine content, and it is suggested that the apparent increase in cystine content is due to the oxidation of tyrosine to 3,4-dihydroxyphenylalanine, which is said to reduce phosphotungstic acid in the presence of sulphite.

J.C.F.

Dimensional Stability of Wool Fabrics

M. Cednäs *J. Textile Inst.*, 52 (June 1961) T251-T271

Three types of shrinkage (in addition to felting shrinkage) can be distinguished when wool fabrics are immersed in water or steamed—(1) A relaxation of strains introduced by previous finishing treatments. The relaxation takes place when the cloth is wetted or treated with wet steam. (2) A change in dimensions which takes place when the moisture content of a relaxed cloth is altered. This change is reversible and is dependent on cloth construction and on the degree of heat-setting given to the cloth. (3) A change in dimensions due to steam pressing. This change can be a relaxation shrinkage, but there is also a specific "press shrinkage" indirectly dependent on the reversible shrinkage.

Usually, yarn in set cloth is stabilised in a curved form. The curvature of set fibres changes reversibly in inverse proportion to their fibre diameters following changes in moisture content. When the equilibrium form of the yarn is changed, the forces of interaction between the warp and weft yarns are changed, and this affects such mechanical properties of the cloth as shear stiffness,

extensibility, and crease recovery. Changes brought about by setting cloth in water for various times at different temperatures and in presence of reducing agents are discussed.

P.G.M.

Setting of Wool Fabrics—a Theoretical Study

B. Olofsson *J. Textile Inst.*, 52 (June 1961) T272-T290

A theoretical framework is given for the experimental results obtained by Cednäs (previous abstract) on the changes in dimensions of set and non-set woven wool fabrics during processing. The resistance to dimensional changes is related to the yarn curvatures. The magnitudes of the dimensional changes during wetting and drying are discussed in terms of change of the set curvature of the yarn. Experimental results are given on the curvature set into hair fibres, and it is shown that the setting results obtained may be explained in terms of an Eyring Flow Element model. It is also shown that a similar model serves to describe the dimensional changes of the set and non-set gaberdine fabrics described by Cednäs.

P.G.M.

Improvement in Stability of Worsted Fabrics

O. A. Nikitina *Tekstil. prom.*, No. 2 (Jan 1961) 54-56

Dimensional changes during the processing of wool mixture fabrics and their influence on fabric shrinkage were investigated under mill conditions. Certain alterations in fabric density, yarn twist (± 100 turns/m) and design were found to have no appreciable effect on the shrinkage of finished fabric when wetted. During continuous processing, machines used after dyeing, i.e. drying and finishing equipment, tend to cause development of excessive tension, resulting in unstable fabrics. Remedies suited to local conditions are suggested.

L.S.L.

Increasing the Felting Properties of Spring-shorn Wool

K. Z. Al'tman *Tekstil. prom.*, No. 3 (Mar 1961) 16-17

Wool from sheep shorn in autumn was found to felt more readily and was preferred for the manufacture of felt boots. The felting power of spring-shorn wool can be increased by a pretreatment (after scouring) for 2 h at 25°C in a bath containing 15 g/l. ferrous sulphate heptahydrate (green vitriol) and 50 g/l. 65% sulphuric acid, at a liquor ratio of 10:1. This is followed by drying at 50-60°C.

L.S.L.

Relaxation Shrinkage of Wool Fabrics—Its Release with Regain and Time

K. Baird *Text. Research J.*, 31 (July 1961) 624-629

Relaxation shrinkage is approximately a linear function of the amount of moisture added to the cloth. The regain must be raised to nearly that of fibre saturation to render any residual shrinkage small. In all work where changes of regain occur, it is most important to distinguish between the effects of relaxation shrinkage and of hygral expansion. When fabric is sprayed with water to remove relaxation shrinkage, the effect is almost complete within 30 min.

C.O.C.

Radioactivity in Wool

R. Kulwich, L. Feinstein, R. Decker, C. Golumbic,

M. Hourihan, and C. Terrill *Nature*, 188 (1960) 511*J. Amer. Leather Chem. Assoc.*, 56 (Mar 1961) 136

The γ -ray activity of clean wool is only a small fraction of that found in greasy wool, which may contain 5-40% dirt. Emission of γ -rays by greasy wool is therefore largely due to the impurities present. It is suggested that γ -ray measurements, which take only a few minutes and are non-destructive, may serve as an index of the amount of impurities present in greasy wool and the relative proportions of the major impurities.

M.T.

Supercontraction of Sound and Weathered Keratin Fibres

O. A. Swanepol *Text. Research J.*, 31 (July 1961) 618-624

Study of the supercontraction of sound and weathered wool and mohair indicates that the second stage of supercontraction results from exposure of sterically protected hydrogen bonds. This can occur by disulphide exchange in the highly cross-linked zones of the microfibrils. Some of the cystine cross-links are stabilised by weathering so that disulphide exchange is limited and hence much less contraction occurs.

C.O.C.

Crystallisation in Wool Keratin

A. Skortchly *Nature*, 191 (8 July 1961) 138-141

A critical review of the literature; 23 references.

C.O.C.

α -Configuration of Fibrous Proteins

R. D. Fraser and T. P. MacRae

Nature, 189 (1961) 572-573*J. Amer. Leather Chem. Assoc.*, 56 (June 1961) 313

The generally accepted concept of the polypeptide chain configuration in the k-m-e-f fibrous proteins (3 α -helices uniformly twisted about a common axis to form a rope) does not agree particularly well with the very wide-angle diffraction pattern obtained from α -keratin and dried muscle. The authors consider that the α -helices may be composed of short straight segments with any distortion required for efficient packing being concentrated at a particular residue or residues, resulting in small but abrupt changes in the direction of the helix axis. Groups of 2, 3 or more such chains might be expected to form segmented ropes of similar average pitch to the uniformly distorted three-strand rope. M.T.

Reaction between Perfluoro-octanoic Acid and the Fibrous Proteins Keratin and Collagen

M. Feughelman and A. Haly

Biochim. Biophys. Acta, 43 (1960) 550*J. Amer. Leather Chem. Assoc.*, 56 (Apr 1961) 190-191

Collagen fibres from rat tail contract rapidly and swell when placed in a satd. aqueous soln. of perfluoro-octanoic acid at room temp. X-ray diffraction photographs of collagen fibres treated for 20 min show a major weakening in high-angle X-ray diffraction pattern. Extended treatment (> 2 days) gives a rubbery material. Apparently the perfluoro-octanoic acid enters into the crystalline structure of the collagen or keratin fibre because of its ability to enter the hydrogen-bonded network which stabilises the crystalline phase. M.T.

Formation of Polymers in Wool

L. J. Wolfram and J. B. Speakman

J.S.D.C., 77 (Oct 1961) 477-482**Structure of Silk Fibres**

A. Wlochowicz

Zesz. Nauk. Politech. Łódz., No. 35

(Wlokiennitwo No. 7) (1961) 59-80

The orientation, crystallite dimensions, and the degree of crystallinity of silk fibres from *Bombyx mori* and *Antheraea pernyi* G. (tussah silk) were investigated by X-ray diffraction. A distinct plate effect was observed in the tussah fibre. The crystallite size was practically the same in both types of fibres, but silk from *Bombyx mori* has a higher degree of crystallinity than tussah silk. T.Z.W.

The Chemistry of Carrotting Processes

E. A. Robinson and J. Robinson

I—Effect of HNO₃ on Rabbit Fur*J.S.D.C.*, 77 (Aug 1961) 351-355**II—Effect of H₂O₂ and HNO₃ on Rabbit Fur***Ibid.*, 356-360**III—Effect of Hg(NO₃)₂ and HNO₃ on Rabbit Fur***Ibid.*, 361-367**Innovations in Artificial and Synthetic Textiles**

P. Herrbach

Teintex, 26 (15 June 1961) 417-429

Review dealing with the following types of fibre—polynosic (cellulosic fibres with microfibrillar structure), Enant (polyamide 7) and Pelargon (polyamide 9), the elastomer Lycra (hydrazine-isocyanate or acid chloride), polyvinyl alcohol, Crylor (acrylic), isotactic poly- α -olefins, graphitic, and fibres from graft copolymers. J.C.F.

Nomenclature for Man-made Fibres

J. E. Ford

J.S.D.C., 77 (Sept 1961) 438-440**Effects of ⁶⁰Co γ -Rays on Viscoelastic Dispersion of Some Textile Materials—I**

K. Sakaoku

J. Soc. Textile Cellulose Ind. Japan, 17 (July 1961) 590-595

With Saran, degradation of the fibre increases with increase in the irradiation dosage, the dynamic modulus decreases, and the dispersion peak of loss-tangent shifts to lower temperatures, with a reduction in the height of the peak. With nylon 6, however, the dynamic modulus increases with irradiation dose up to 5×10^7 rep (at room temperature), but shows a decrease at 10^8 rep when compared to non-irradiated material. It is concluded that with an irradiation dose up to 5×10^7 rep the cross-linking effect predominates in nylon 6, but that at 10^8 rep degradation takes place. Crystalline polyethylene terephthalate exhibited an increase in dynamic modulus with irradiation dose up to 10^8 rep, and the peak of loss-tangent,

which appears at the glass-transition temperature, barely changed. It is considered that irradiation has an annealing effect, and that there is no relationship between cross-linking and degradation. I.G.L.

Effect of Cold Drawing on Orientation and Crystallinity of Polycaprolactam Fibre

G. W. Urbanczyk

Zesz. Nauk. Politech. Łódz., No. 35

(Wlokiennitwo No. 7) (1961) 141-162

As the draw ratio changes, fibre texture (appearance of plate effect), crystallinity, and the average crystal size also change. The highest crystallinity and greatest average crystallite size are found at 315% effective elongation. The total increase of crystallinity in comparison with the undrawn fibre is 20-25%. T.Z.W.

Absorption of Moisture by Polycaprolactam Fibre

E. Turska and J. Wiśniewski

Zesz. Nauk. Politech. Łódz., No. 35

(Wlokiennitwo No. 7) (1961) 45-57

Absorption of moisture by unstretched polycaprolactam fibre was determined. At constant temp. the rate at which the absorption equilibrium is reached is inversely proportional to the relative humidity. This rate is greatest initially and decreases as equilibrium is approached. Absorption of water causes elongation of the fibre, but this ceases long before equilibrium is reached. At high relative humidities, after initial stretching, partial contraction of the fibre is observed. T.Z.W.

Mass-pigmented Nylon Yarns

G. K. Mecklenburgh

B.N.S. Outlook, No. 10 (Spring 1961) 2-7

A general review of the properties of the pigments used, their preparation, and the colouring technique. The properties of the coloured yarns are also considered. M.T.

Terylene Bulk Yarns

C. S. Bruce

J. Textile Inst., 52 (Aug 1961) P371-P397

This paper, which was delivered at the Textile Institute Conference on "Bulked Yarns and other Modified Textiles" describes the production of false-twist, stabilised false-twist, Ban-Lon, and Taslan yarns from Terylene, their properties, and uses. False-twist Terylene yarns have been used successfully where stretch is a prime requisite, mainly in woven structures, and in order to develop the full yarn bulk it is necessary to scour at the boil. In order to avoid permanently set creases the fabric should be treated either in book form or in a Hennekin continuous scouring unit where the cloth is moved in one direction in open width whilst the scouring liquid is circulated in the opposite direction. W.G.C.

Polymer Deposition in Polyvinyl Alcohol Fibres

K. Matsubayashi

I—Polymer Deposition in Wet-spun Fibre, Heat-treated Fibre, and Various Acetalised Fibres*J. Soc. Textile Cellulose Ind. Japan*, 17 (June 1961) 498-502

The deposition of polymer in wet-spun fibre, heat-treated fibre, and acetalised fibre was investigated. The amount of polymer deposited was increased by acetalisation with glyoxal. The free aldehyde groups thereby introduced into the fibre were found to participate in redox polymerisation and, further, graft polymer is considered to be produced as a consequence of chain initiation by carbonyl radicals produced in the glyoxalised fibre. The effect of glyoxalisation on the amount of polymer deposited is therefore considered to be due to the free aldehyde groups introduced into the fibre. The quantity of polymer deposited is also increased by acetalisation with benzaldehyde and tetrahydrobenzaldehyde. Hot-drawing and heat-treatment, on the other hand, reduce the amount of polymer deposited in the fibre.

II—Properties of Polyvinyl Alcohol Fibres containing Deposited Polymer*Ibid.*, 502-507

Polymer deposition results in a decrease in tenacity, slight increase in elastic recovery, and improved heat-setting properties. The heat-setting properties and elastic recovery of benzalised fibres containing polyacrylonitrile are superior to those of formalised fibres containing polyacrylonitrile. The dry-softening temperature is lowered on deposition of polystyrene, but is hardly affected by deposition of polyacrylonitrile. The effects of

polymer deposition on the thermal properties of the fibre are considered to be largely dependent on the thermal properties of the polymer itself. I.G.L.

PATENTS

Dye-receptive Graft Copolymers

Dow Chemical Co. BP 876,001 (U.S.A. 29 June 1958)

Polyesters grafted with ethylenically unsaturated monomers, e.g. by swelling the polyester and then polymerising the monomer *in situ* in the polyester, are readily dyed with various classes of dyes, depending on the monomer used. Thus use of *N*-vinylpyrrolidone confers affinity for direct, acid, and disperse dyes. C.O.C.

Polysulphonamides

ICI

BP 875,067 (25 Nov 1957)

An aqueous solution of a polymethylene diamine (1 mol.) is mixed with a solution of an aryl disulphonyl halide (1 mol.) in a water-immiscible organic solvent in presence of < 2 mol. of a strong base in excess of that required to neutralise the hydrogen halide formed during the reaction at 20–100%. Thus the system is always in the liquid phase. The resulting aqueous solution of the polysulphonamide can be used directly, e.g. to form fibres by extrusion.

BP 875,068 (25 Nov 1957)

A solution containing both *p*- and *m*-substituted aromatic polysulphonamide is extruded into a coagulating bath with which the solvent is miscible in all proportions.

BP 875,069 (25 Nov 1957)

An aqueous coagulating bath of pH less than that of the polymer solvent is used.

BP 875,070 (25 Nov 1957)

A polysulphonamide having recurring units of formula $-SO_2R^1SO_2NR^2R^3NH-$ (R^1 = aromatic; R^2 = H or Alk; R^3 = polymethylene) is dissolved in a mixture of $< 80\%$ by wt. of an alcohol, 0–20% of water, and $> 20\%$ of a strong base.

BP 875,071 (25 Nov 1957)

Dimethylformamide is used as the solvent for the polysulphonamide.

BP 875,072 (25 Nov 1957)

The polysulphonamides have much improved resistance to alkali if they contain a high concentration of *N*-substituted sulphonamide groups. C.O.C.

Effect of Sulphuric Acid on Wool. V—Investigation of Dyeing Faults caused by Carbonising (VII this page)
Solvent-extraction of Detergent-scoured Wool (VII this page)

Mass-coloured Polypropylene (VIII p. 583)

Crystalline Polyolefins having High Affinity for Dyes (XIII p. 591)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Tensile Behaviour of some Cellulose Rayons Swollen in Sodium Hydroxide Solutions

B. Farrow *J. Textile Inst.*, 52 (May 1961) T221–T240

Extensometric data are presented for four commercial rayons—ordinary viscose rayon, Tenasco, Durafil, and Fortisan—swollen in NaOH soln. of concn. up to 7*N*, at 0–80°C. Length changes on immersion of samples free from load are also recorded. Length changes are the resultant of (i) contraction, by partial release of long-chain molecules originally held in extended form, (ii) contraction, by release of cross-bonding in a strained network, (iii) elongation, by the forcing apart of molecular parts by the swelling agent. With a high degree of swelling the net effect is contraction for highly orientated rayons and extension for those of low orientation. The behaviour of the rayons when swollen depends on the method of forming and stretching the fibres rather than on the D.P. of the cellulose. With Durafil and Fortisan, which have a single and strong system of bonds, the swelling agent weakens but does not disrupt the structure, and breakage of the swollen fibre is by molecular scission. Tenasco and ordinary viscose rayon have two systems of bonds which together afford only moderate resistance to swelling forces, and when the swollen fibre is extended, breakage is chiefly by molecular slippage. A study of load-extension behaviour and dimensional changes in alkali should enable differences in fine structure to be detected even with

rayons which differ only slightly in accessibility and crystalline ratio. W.K.R.

Bleaching of Textiles—Science and Art of Bleaching

G. A. Langer *Canadian Textile J.*, 78 (7 July 1961) 41–44

Some of the bleaching techniques in general use, as well as several recent developments, are discussed. Data are presented on the uses of hydrogen peroxide, sodium chlorite, potassium permanganate, sodium hypochlorite, and sodium hydrosulphite as used in the bleaching of cotton, wool, and synthetic-fibre blends. S.B.D.

Action of Hydrogen Peroxide on the Tryptophan of Wool

P. Miro

Bull. Inst. Text. France, (94) (May–June 1961) 65–71

The tryptophan contents (colorimetric—*p*-dimethyl aminobenzaldehyde) have been determined of samples of wool, untreated and after treatment with various concentrations of H_2O_2 , of the same samples after the treatment with 6*N* HCl necessary for the isolation of cortical cells, and of the ortho- and para-cortical cell fractions subsequently isolated. The results indicate that the tryptophan in wool can be divided into two fractions—(a) that in the intracellular material, which is very accessible to chemical attack, and (b) a more resistant fraction located within the cortical cells. The present evidence suggests that the tryptophan contents of the ortho- and para-cortical cells of untreated wool are the same. J.C.F.

Effect of Sulphuric Acid on Wool. V—Investigation of Dyeing Faults caused by Carbonising

R. L. Elliott, R. S. Asquith, and B. J. Jordan

J.S.D.C., 77 (Aug 1961) 345–349

Solvent-extraction of Detergent-scoured Wool

R. G. Stewart *J. Textile Inst.*, 52 (May 1961) T241–T247

Raw wool was scoured with a non-ionic detergent—to avoid contamination with soap—and then Soxhlet-extracted with various solvents. The quantity of extract differed widely according to the solvent used, and some extracts contained a considerable amount of fatty acid. It is suggested that the substance held most tenaciously by the fibre has a high fatty-acid content. The extractability of the fatty matter was reduced by oven-drying the wool before extraction. Dry wool gave a lower extract than conditioned wool, and conditioned wool gave a higher extract when previously dried at a low temp. than when dried at high temp. The results favour the view that water in conditioned wool assists in fat removal by emulsification, and do not support the “soap barrier” theory. Light petroleum is considered to be as satisfactory as diethyl ether as a solvent for the determination of fatty matter in scoured or greasy wool. W.K.R.

PATENTS

Continuous and Rapid Bleaching of Cotton Cloth

DuP

USP 2,960,383 (25 July 1958)

A full white is obtained in < 1 h by saturating the cloth with an aqueous solution containing 2.5–6.0% NaOH (cloth containing size needs 4–6%) and tetrasodium pyrophosphate, Na hexametaphosphate, and Na tripolyphosphate, keeping the saturated cloth for 2–10 min at 200–212°F (preferably 5–8 min at 205–212°F), and removing the caustic soda. It is then saturated with an aqueous solution containing, by wt., 0.0–6% NaOH, 1–3% Na silicate (42°Bé), and 0.5–1.2% H_2O_2 , and kept for 2–10 min at 200–212°F (preferably 5–8 min at 205–212°F), rinsed, and dried. C.O.C.

Chlorite Bleaching of Cellulosic Material

Bleachers Assoc.

BP 875,020 (25 Mar 1958)

Bleaching with chlorite can be carried out at high pH and in absence of a buffer if monomethyloldimethyl hydantoin or a dimethyl hydantoin-formaldehyde resin is present. Thus spun rayon (5 g) was, without desizing, boiled for 30 min in a solution containing, per litre, 10 g Na chlorite and 1.25 g monomethyloldimethyl hydantoin, the liquor ratio being 100:1. The pH was 7.8 at the start and 8.0 at the finish. A full white was obtained having a reflectance of 90%. C.O.C.

Bleaching Mechanical Wood Pulp with Boranes

DuP

USP 2,960,427 (3 Sept 1958)

Primary, secondary, and tertiary amines having a borane (BH_3) substituent on the N atom and ammonia-borane $H_3N:BH_3$ are effective bleaching agents for pulp

slurries of either low or high (1–20%) consistency. They can be used at any temperature at pH 3–14, but are preferably used at 40–80°C and pH 5–11. C.O.C.

Bleaching Lignocellulosic Pulps with Hydrosulphites

Crown Zellerbach Corp. *USP* 2,963,395 (31 Mar 1958)
Pulp containing 15–50% by weight of dry fibre has the air removed from it, is mixed with an aqueous solution of a hydrosulphite and a stabiliser, and is then kept at 100–230°F in absence of O₂ and at pH 4–8, the pulp consistency being maintained at 15–50% until the desired brightness is obtained. C.O.C.

Peroxide Bleaching of Linoleum

Armstrong Cork Co. *USP* 2,960,382 (13 May 1955)
The linoleum is coated with an aqueous solution containing 35% by wt. of H₂O₂ and, in addition, a strong alkali, e.g. NaOH or Ca(OH)₂, a stabilising agent, e.g. Na silicate, and a wetting agent. It is then immediately dried by passing it at 100 ft/min through a chamber at 230–270°F. As it leaves the chamber it is sprayed with dilute acid, e.g. 1% H₂SO₄, rinsed, and dried. This procedure prevents any damage to the binder or base sheet and is carried out at normal production speeds. C.O.C.

Wet Processing of Textiles in a Fluidised Bed (VIII p. 583)
Rendering Permeable Materials Bacteriostatic (X p. 587)

VIII—DYEING

Continuous Dyeing

H. Bors *Chemiefasern*, 11 (Aug 1961) 544–550
The fields in which continuous dyeing is finding application, and those in which successful methods have yet to be developed are reviewed briefly, as are the general advantages and disadvantages of the method. Factors contributing to the success of the process are plant design, the dye used (lowest possible affinity) and the material itself (maximum uniformity, freedom from size or other residues, easily wettable). A universally applicable installation is discussed, and methods of applying direct, reactive, vat, solubilised vat, azoic and sulphur dyes are given. Some commercially available continuous processes are reviewed. I.G.L.

Physico-chemical Properties of Dye Solutions and the Adsorption of Dyes on the Fibre. III—Relation between Activity Factor and Adsorption of Methyl Orange

M. Mitsuishi and G. Aida *J. Soc. Textile Cellulose Ind. Japan*, 17 (July 1961) 665–669
The solubility of Methyl Orange (C.I. 13025) has been determined at 80, 60, 40, 20, and 0°C in absence and in presence of 0.1–1.6 mole/l. of NaCl. The activity factor ($f.k_a^{-1}$) has been calculated from the equation

$$f.k_a^{-1} = ([Na^+][D^-])^{-1}$$

(where f = mean activity coefficient of dye solution, k_a = solubility product, and $[Na^+]$ and $[D^-]$ = sodium and dye ion concentrations, respectively). Activity factors (at 80, 60, 40, 20, and 0°C) and amounts of dye adsorbed by Cellophane (at 80 and 60°C) have been determined over the range of salt concentrations given above. It is concluded that—(1) $f^{-1}.k_a$ is proportional to the sodium ion concentration, (2) amount of dye adsorbed on direct dyeing of Cellophane sheet is proportional to $f^{-1}.k_a^{-1}$. I.G.L.

Constitution and Application of Vat Dyes

E. R. Schober *Canadian Textile J.*, 78 (4 Aug 1961) 37–41
A brief history of vat dyes and their chemistry is presented, together with data on the amounts of hydrosulphite and caustic soda required in their application and the importance of these compounds in obtaining level dyeing and satisfactory fastness. Information on pigmentation methods is also presented. S.B.D.

Use of Alkaline Cellulose Sulphite in Dyeing with Solubilised Vat Dyes

V. V. Pashkevichyus and I. I. Putnaite *Tekstil. prom.*, No. 2 (Jan 1961) 72–73
The use of cellulose sulphite as a protective colloid in one-bath application of solubilised vat dyes is suggested. The assistant is said to prevent the precipitation of loose oxidised dye on to the fabric. For concn. of up to 0.4 g/l.

of dye, 0.5 g/l. of assistant is suggested; at higher dye concn. 1–2 g/l. of assistant must be used. Detailed recipes for certain colours are given. L.S.L.

Yarn Dyeing with Phthalogen Dyes—II

G. Textor *Textil Praxis*, 16 (June 1961) 591–592
In hank dyeing the yarn is mordanted at 80°C with the total amount of Phthalofix FN for 10 min without salt; sodium sulphate is then added in two portions and treatment continued for 20–30 min. For pale depths, 0.5% Levegal KN or Naphtopon E assists levelling. After thorough cold rinsing, the yarn is introduced into a bath containing acetic acid, Naphtopon E and, if necessary, Trilon B, and the dye is then added in one portion. Good circulation is required during the 30–45 min dyeing. Additional Naphtopon E assists penetration of high-twist yarns. After two cold rinses the dye is developed by treating the goods for 15 min at 60°C in the prepared developing bath. About 25% less dye is required to achieve a given depth on mercerised yarn. Cupressa yarns can also be dyed with the standard machines and technique, but to preserve the bulk of Cupressa yarns only vat- or hank-dyeing machines are recommended. Salt is not used in mordanting, however, owing to the high affinity of these two yarns for the mordant. In the package dyeing of flock the final soap treatment is followed directly by spin-drying and a hot 0.1% soda rinse. With fuller depths a second soap bath is often necessary to remove unfixed pigment particles. In dyeing warp beams the weight should not exceed 70 lb to avoid levelling and penetration difficulties. Single-bath bleaching and mordanting is an advantage since the yarn swells less and is softer than with successive bleaching, rinsing, and mordanting. In dyeing silk, the degummed and rinsed yarn is mordanted in the usual way, introduced into the acetic acid/Naphtopon E bath, and the dye added in one or two portions. After dyeing cold for 30 min, the yarn is rinsed well and developed with Decrolin Soluble Conc. and formic acid at 80°C for 20 min, followed by rinsing and soaping.

Phthalogen dyes are not recommended for bleachable materials, since chlorine bleaching at low pH can degrade the metal-dye complex and the released metal can lead ultimately to fibre damage. Relevelling of faulty goods in a blank vat is not possible, stripping and redyeing being necessary to correct defects. Stripping is effected with 0.5–1.0 g/l. potassium permanganate and 0.5–0.6 ml/l. sulphuric acid (66°Bé) in a cold bath for 30 min, followed by a rinse and cold-treatment with 4 ml/l. sodium bisulphite liquor and 2 ml/l. formic acid (85%) until manganese oxides are removed. It may be difficult to obtain good stripping with hank yarns and cheeses, and because of this and possible fibre damage, stripping is to be avoided. Experience shows that Phthalogen dyeings can be carried out in 4 h on standard equipment and that costs are similar to those of the average Indanthrene dyeings. Yarn penetration is excellent and uniformity of dyeing on mixed cotton-viscose rayon yarns good. E.C.

Producing Olive Green Dyeings without use of Conventional Synthetic Dyes

P. S. Nadkarni *Defence Sci. J. (New Delhi)*, 10 (1960) 320–325
Chem. Abs., 55 (7 Aug 1961) 15936

Attempts were made to produce olive greens on textiles by using mixtures of inorganic pigments with or without Aniline Black (C.I. Oxidation Base 1) and Bhilawa Nut Black for comparison of cost and fastness properties with standard Mineral Khaki-topped vat dyeings on service clothing. Some black pigment was necessary to give the depth of colour required, and Lamp Black (C.I. Pigment Black 6) with a binder Bedafin 2101 (ICI), used with PbCrO₄ (C.I. 77600), gave good fastness to light but poor fastness to washing. Aniline Black in the small amounts required is not fast to light. C.O.C.

Reactone Dyes in Dyeing and Printing

H. Schumacher *Teinture et Apprête*, (63) (Apr 61) 55–63
Reactone dyes (Geigy) are a new series of reactive dyes for cellulosic fibres, based on tetrachloropyrimidine. This review describes typical recipes for the principal dyeing techniques, tests of washing fastness and substantivity, printing formulations and methods, and the use of Reactone dyes in conjunction with resin finishes. J.C.F.

Bond between Reactive Dye and CelluloseO. A. Stamm, H. Zollinger, H. Zähler, and E. Gümman
Helv. chim. Acta, **44** (15 June 1961) 1123-1125

The covalent nature of the bond between a reactive dye and the cellulose mol. is established by microbiological degradation of the dyed cellulose and identification of glucose in the hydrolysate of a dyed mol. degradation product. The reaction of glucose with a Procion dye leads to a cpd. from which glucose may be recovered by acid hydrolysis.

H.H.H.

The Combination of Reactive Dyes with Cellulose

H. Rath and M. Passler

Melliand Textilber., **42** (July 1961) 787-791

An attempt is made to show that monochlorotriazine dyes and cellulose are linked by a covalent bond. Model dyes are prepared and the infrared absorption of their dyeings on cellulose powder is compared with that of simple alkoxy compounds prepared from the dye. The spectra obtained are complicated and indicate, but do not prove, the formation of a C-O-C bond.

P.B.S.

Causes of Tailing on Application of Drimarene Z Dyes by the Cold Pad-Batch ProcessM. Capponi *S.V.F. Fachorgan*, **16** (May 1961) 341-351

In the application of Drimarene Z dyes by this method, substantivity of the dye and reduction of hydroxyl ion concentration of the pad liquors during the process are considered to be the most likely causes of tailing, and their relative importance has been investigated. It is concluded that tailing is not a substantivity effect, but is caused solely by the reduction in OH-ion concn. during padding when caustic soda alone is used as alkali in the pad liquor. Tailing can, however, be practically eliminated by using a caustic soda-sodium metasilicate mixture in place of NaOH.

I.G.L.

The Single-bath Padding Batching Process for Remazol Dyes

H.-U. von der Eltz and K. Wagner

S.V.F. Fachorgan, **16** (May 1961) 320-340

The method is reviewed in detail and its advantages are demonstrated. Practical details and experience gained in two years' application are also discussed. The effects of different alkalis and alkali concentration, and factors influencing the rate of fixation, have been investigated. The fixation rate is higher the stronger the alkali used. At temperatures above 25°C the fixation rate increases with increase in fixation temperature. Increase in dye or electrolyte concn. results in a higher fixation rate. Additions of salt improve the yield with certain dyes. Fixation takes place more rapidly on regenerated cellulose than on cotton. Comparisons of stability and fixation rate have also been made—comparing in five cases half-life periods of padding liquors with half-dyeing times. Ratios of the former to the latter have been calculated as a rough indication of the relative speeds of reaction of the dye with the fibre and with water.

I.G.L.

Azoic Dyeings using Chloronaphthylamines

H. F. Bassilios, M. Shawky, and A. Y. Salem

J. Chem. U.A.R., **3** (1960) 251-264*Chem. Abs.*, **55** (21 Aug 1961) 17007

Azoic dyeings on cotton were made using the seven possible isomeric 1-chloronaphthylamines as diazo components and Naphtol AS-SW (C.I. Azo Coupling Component 7), and their fastness properties determined. The visible absorption spectra of the dyes are given. C.O.C.

Adsorption of Direct Dyes by Chemically Modified Celluloses

E. H. Daruwalla, P. J. Kangle, and G. M. Nabar

Text. Research J., **31** (Aug 1961) 712-721

Equilibrium adsorption of Chrysophenine G (C.I. Direct Yellow 12) and Chlorazol Sky Blue FF (C.I. Direct Blue 1) on cellulose oxidised with potassium metaperiodate, dichromate-sulphuric acid, dichromate-oxalic acid, and sodium hypochlorite, and on their corresponding borohydride-reduced products and sodium-chlorite-treated samples were determined under standard conditions of dyeing. Conversion of hydroxyl groups at 2, 3, or 6 position in the glucose units of the cellulose did not alter the uptake of dye, but formation of carboxyl groups reduced it markedly. The extent of reduction in dye

adsorption at a specific stage of oxidation and electrolyte concentration in the bath is the same irrespective of the position in the glucose unit at which the carboxyl group has been formed. Only a small fraction of the total carboxyl groups is responsible for the reduction in dye adsorption, and with progressive increase in the total carboxyl content the value of this portion continuously decreases.

C.O.C.

Influence of Sulphur Dyes on Strength of Cotton Fabric

J. L. Khmel'nitskaya and Z. I. Sergeeva

Tekstil. prom., No. 2 (Jan 1961) 52-54

Samples of cotton fabric were dyed with a range of sulphur dyes and subjected to an accelerated ageing test. At intervals the samples were examined for strength, copper number, pH of water extract and presence of $-SO_3^-$ ions in the water extract. Blue and green sulphur dyes did not affect the fabric strength and there was only a slight change in shade (redder). Certain browns—Immedial Dark Brown A (C.I. 53245) and Sulphur Brown were found to have a similar action to Sulphur Black (C.I. 53185), i.e. degradation of dyed sample and an increase in the acidity of the water-extract were observed. This behaviour was explained in terms of the oxidation of disulphide and polysulphide linkages of the dye and formation of sulphuric acid. With the above-mentioned browns the rate of acid development was rather low and should have no practical effect on the properties of the fabric.

L.S.L.

Dyeing Cotton with Aniline Black

E. Trepke and C. Burski

Zesz. Nauk. Politech. Łódz., No. 33 (Chemia 9) (1961) 103-111

Conditioned cotton samples dyed with Aniline Black are dissolved in 80% H_2SO_4 . The soln. is left standing at room temp. for 72 h. It is then suitably diluted with 80% H_2SO_4 and its optical density, and that of the solvent, is measured, to obtain the amount of Aniline Black on the fibre. Addition of a large excess of free aniline in the dye-bath does not decrease acid tendering of the cotton when it is dried at 40-50°C but is beneficial at 60-70°C. The low drying temp. causes an increase in the amount of dye on the fibre. The replacement of aniline hydrochloride by the formate decreases the extent of tendering and the amount of dye formed. The best results are obtained with 90% hydrochloride, 10% formate, 3 g/l. aniline in the padding liquor, and a drying temp. of 70°C.

T.Z.W.

Application of Pigments to Fabric

G. G. Petzshik, E. Ya. Davydova, and L. V. Lezneva

Tekstil. prom., No. 3 (Mar 1961) 44

The following procedure is suggested for the application of phthalocyanine pigments to cotton-synthetic-fibre fabrics. Material is padded in a bath containing (per 1000 parts)

Latex S.V.Kh. (aqueous emulsion of 40% polyvinylidene chloride, 60% polyvinyl chloride)	60
Preparation OP 10 (polyethylene glycol ether of iso-octaphenol)	8-5
Pigment	20
Olikazin (thermosetting resin)	24-6
Ammonium chloride	2-0
Sodium alginate (6% solution)	22-3
Soda	2-3
Water	881-3

After padding, the fabric is dried and baked at 140°C for 5 min. Washing-off is unnecessary.

L.S.L.

Fast Dyeing of Wool-Cellulose Fibre Mixtures

D. R. Lemin and J. K. Collins

S.V.F. Fachorgan, **16** (July 1961) 470-482

An account of dyeing with various classes of dyes. Reactive, ingrain, vat, and azoic dyes are the most suitable. Various dyeing methods for each class and details of the faults associated with the dyeing of these mixtures are given. It is shown that level dyeing depends upon the careful choice of suitable dyes and conditions.

P.B.S.

Levelling Problems in Wool DyeingJ. G. Graham *S.V.F. Fachorgan*, **16** (July 1961) 464-470

Experiments are described which show the value of Dispersol CWL as a levelling agent for Carbolan and

Coomassie dyes. An account is given of the influence of this agent on other dyes and on practical dyeing methods. P.B.S.

Simplified Method of Wool Dyeing

N. L. Baskin, N. I. Podgornaya, A. A. Grigor'eva, and N. S. Gerasimova *Tekstil. prom.*, No. 2 (Jan 1961) 70

A new restraining agent, Sulfonol NP-1, has been found to be satisfactory in the dyeing of wool, both as loose fibre and as fabric. It is suggested that for 0.2–0.5% of dye, 0.5–4% sulphuric acid and 0.5 g/l. Sulfonol NP-1 should be used. The assistant can also be used at 1.0 g/l. in the dyeing of carbonised wool without neutralisation. It has been also found to be useful in the dyeing of Kapron. L.S.L.

Dyeing Woollen Yarn in Oil

G. A. Smith *J.S.D.C.*, 77 (Sept 1961) 438

Improvement of the Light Fastness of Dyes and Dyeings

H. Rath and H. J. Brielmaier

Melliand Textilber., 42 (Aug 1961) 911–912

Dyes, when exposed to light, are in an excited state and so more susceptible to the reactions leading to fading. Experiments are described in which fluorescence quenching compounds, which convert light energy to kinetic energy, are linked to the fibre by covalent bonds, thus absorbing the light energy causing fading. Increased light fastness of basic and acid dyes on wool was achieved. P.B.S.

Reactive Dyes for Man-made Fibres

R. L. Denyer and J. A. Fowler

Melliand Textilber., 42 (May 1961) 535–540

The application of Procion dyes to regenerated cellulose fibres is first discussed. In the application of cold-dyeing dyes, sodium bicarbonate may be used at 50–60°C instead of sodium carbonate at 20–30°C. The warm bicarbonate method is advantageous for certain dyes, and particularly for high-denier fibres and tightly woven fabrics. Differences in behaviour in the two methods are interpreted in terms of rates of diffusion and degree of aggregation of the dye in the dye bath. The dependence of fixation (%) on pH, temperature, and, in the case of viscose rayon staple, denier on application by the warm bicarbonate technique is illustrated. Cold-dyeing dyes for viscose rayon have been classified as (i) giving fuller depths, (ii) giving similar depths, and (iii) giving paler depths by the bicarbonate technique than by use of Na₂CO₃ at 20–30°C. Semi- and fully-continuous methods for cold-dyeing dyes are reviewed briefly, as are the properties and methods of application of the hot-dyeing Procion dyes. Both types of dye can be applied to cotton by the Procion-Resin Process. Difficulties in application to viscose rayon are discussed. Application of Procynyl dyes to polyamide fibres is reviewed, and the mechanism of dyeing is discussed. The dependence of fixation and dyebath exhaustion on pH and temperature is illustrated, and optimum conditions for application to nylon are given as—Stage 1: pH 3.5–4 at 95°C for 30 min (minimum). Stage 2: pH 10 (using sodium carbonate) at 95°C for 2 h. Fixation of dye continues after the amino end-groups have reacted, although more slowly. When dyeing is continued thus the nylon retains its tensile strength and extensibility, showing that no new primary amino end-groups have been formed by hydrolysis of amide groups. Additional uptake of dye is therefore considered to be due to reaction of the dye with the amide groups. Confirmation that this reaction is possible has been found. I.G.L.

Acrylic Fibres—Dyeing Progress

R. Sureau *Textintex*, 26 (15 June 1961) 401–417

After a brief review of earlier processes employing conventional dyes, with or without special modifications of the dyeing process (cuprous ion, hydroxylamine), the author deals at length with basic dyes, including the newer basic anthraquinone and quaternary heterocyclic azoic dyes. 9 References. J.C.F.

Studies on the Dyeing of Acrylonitrile Fibre

IX—Purification and Estimation of Acid Dyes

Y. Takase *J. Soc. Textile Cellulose Ind. Japan*, 17 (June 1961) 526–532

Purification (mainly the removal of inorganic material and intermediates) of a large range of azo acid dyes has

been investigated, salting-out with sodium acetate and the use of a weakly basic ion-exchange resin being compared. The former was preferable. Dye strengths were estimated by titration with titanous chloride.

X—Conditions of Application of Azolic Coupling Components to Acrylonitrile Fibre

Y. Takase and T. Ogawa *Ibid.*, 532–536

Absorption by acrylonitrile fibre of β -naphthol (I), 2,3-dihydroxynaphthoic acid (II), Naphthol AS (III), and Naphthol AS-BO (IV) has been studied. III and IV proved unsuitable because of their low rates of diffusion, but I and II can be applied as coupling components. The absorption of II, and to a lesser extent of I, is dependent on the pH—decreasing as the pH rises owing to the formation of alkali-metal salts. Variation of exhaustion with time at various pHs and temperatures (70, 80 and 90°C) has been investigated, and it is concluded that the most suitable conditions for application of I and II are a bath temperature of 98°C, liquor ratio 50 : 1, pH < 3–4, and time 60–90 min. I.G.L.

Dyeing Conditions and Practical Saturation Value of Disperse Dyes on Polyester Fibres

J. Carbonnel *Teinture et Apprêts*, (64) (June 1961) 91–104

Reduction of the amount of dispersing agent gives a higher degree of exhaustion and better rubbing fastness of the resulting dyeings, although there is a slight impairment of levelling if dyeing times are short. For very long dyeing times the amount of dispersing agent can be reduced still further. If the liquor ratio is greater than 40 : 1, the amount of dispersing agent required should be calculated on the weight of fibre, and not on the bath volume. J.C.F.

Effect of Organic Auxiliaries in Dyeing of Polyester Fibres with Disperse Dyes

E. Elöd and Vinh-Am

Textil Praxis, 16 (July 1961) 718–724

Physico-chemical data on the adsorption of the 9 disperse Celliton dyes (C.I. 10345, 11005, 11110, 26090, 61100, 60700, 60710, 61500, and 61505) are reported, and the influence of dye structure and diffusion discussed. The mechanism of adsorption by polyglycol phthalate fibres, alone and in presence of carriers (benzyl alcohol, *m*-cresol, phenylmethyl carbinol, diphenyl ether, dimethyl terephthalate, benzoic acid, diphenyl ketone, dimethyl phthalate, *o*-dichlorobenzene, *o*-phenylphenol, and methyl salicylate) is discussed, and the colour behaviour of the above dyes in the adsorption process in the presence of carriers described. 21 References. H.H.H.

II

Ibid., 16 (Aug 1961) 797–802
An account is given of an investigation into the influence of carriers and their concn. on the affinity of disperse dyes for polyester fibres, and on the stress-strain behaviour, swelling properties, moisture absorption, and specific gravity of the fibres. J.W.D.

Light Fastness of Basic Dyes on Acid-modified Dacron

S. B. Maerov and H. Kobas

Text. Research J., 31 (Aug 1961) 697–703

The Critical Fading Region (c.f.r.) of basic dyes on acid-modified polyethylene terephthalate fibres is a rather narrow spectral region extending from 350 to 425 m μ . Substituted hydroxybenzophenones, e.g. 2,2'-dihydroxy-4,4'-dimethoxybenzophenone (I) act as light screeners with many basic dyes in the c.f.r. and increase the fastness to light of their dyeings. Many of these screeners can be applied by normal dyeing techniques. The requirements for efficient screening ability of these agents are discussed. Photosensitisation of dyes by some screeners with resultant diminution of light fastness is ascribed to radiationless transfer of energy from screener to dye by a resonance transfer mechanism. Chemically modified I gave improved light fastness to deep dyeings of triphenylmethane dyes. The improvement in light fastness is caused by the dye's enhanced bathochromic absorption in the c.f.r. relative to the absorption spectrum of I. C.O.C.

Dyeing of Polyvinyl Chloride Fibre

E. P. Friess *S.V.F. Fachorgan*, 16 (July 1961) 442–447

The properties of several types of commercial polyvinyl chloride fibres are listed and the difficulties of dyeing

polyvinyl chloride and ways of overcoming or lessening these are discussed. A detailed account of the methods of dyeing with disperse, azoic, and metal-complex dyes ends with a brief survey of the patent literature. P.B.S.

Leather Dyeing using Acetone as Solvent

L. Nagel and M. A. D. Carlo

Rev. fac. cienc. quim. Univ. Nacl. La Plata,
32 (1960) 121-130

Chem. Abs., 55 (24 July 1961) 14954

Replacing water by acetone increases the solubility of the dye and the rate of dyeing. The resultant dyeings differ in no respect from those obtained in aqueous dye-baths. C.O.C.

Dyeing Anodised Aluminium and its Alloys

N. N. Abramova

Zashchito-Dekorativ. i Spetsial. Pokrytiya Metal.,
Nauch.-Tekh. Obshchestvo Mashinostroitel. Prom.,
(1959) 123-130

Chem. Abs., 55 (24 July 1961) 14116

Application of azo dyes to aluminium that has been electropolished and anodised using H_3PO_4 - H_2SO_4 - CrO_3 and 15-20% H_2SO_4 , respectively, is discussed. C.O.C.

PATENTS

Wet Processing of Textiles in a Fluidised Bed

Shri Ram Institute for Industrial Research

BP 874,232 (India 3 Nov 1956)

The impregnated fabric passes through a fluidised bed of solid particles of size 1600-160 μ at 120-170°C for > 5 s and then immediately into a chamber at 80-105°C for 10-60 s. This ensures that the chemical reaction, e.g. of dyes or resins, is carried out in moist conditions while permitting sudden application of heat. The moisture content of the cloth can be controlled throughout, and degradation of the fibre or the chemicals, e.g. Na hydro-sulphite or leuco vat dye is prevented. C.O.C.

Dyeing Cellulose with Sulphur Dyes

CFM

BP 874,151 (Germany 28 Feb 1959)

Alkali-metal sulph-hydrates are good reducing agents for the water-soluble thiosulphonic acids of sulphur dyes. Thus thiosulphonic acid of Immedial Brown BR (C.I. 53270) (made by the process of GP 91720) (80 g) is dissolved in water (20 l.). Aqueous 22% Na sulph-hydrate (120 ml) and Na_2SO_4 (40 g) are added, and spun viscose rayon fabric (1 kg) is dyed in the liquor for 1 h at 80-90°C. The resultant dyeing has the same hue and depth as one obtained using Na_2S as the reducing agent, and the dyed fabric has an excellent handle. C.O.C.

Vat Dyeing or Printing Blends of Cellulose Fibres and Fibres of Cellulose Esters of Low Hydroxyl Content

Celanese Corp. of America USP 2,961,288 (23 Feb 1955)

The fibres are padded or printed with a vat dye and then heated to cause the dye to penetrate into the cellulose ester. They are next treated with a reducing agent to cause dyeing of the cellulose and finally oxidised and soaped in the usual manner. C.O.C.

Solid Dyeings on Cellulose-Protein Fibre Blends with Reactive Dyes

ICI

BP 875,163 (28 Sept 1959)

Uptake of cold-dyeing reactive dyes, e.g. those described in BP 785,222 (J.S.D.C., 74 (1959) 58) can be retarded by adding an organic compound containing an anionic water-solubilising group to the dyebath, e.g. the Na salt of sulphonated methyl oleate. This enables dyeing of the protein fibre to be controlled so that the fibre is dyed to the same or less depth than the cellulose. C.O.C.

Dyeing and, if desired, Simultaneous Permanent Waving of Hair

N.V. Industriële Onderneming W. H. Braskamp

BP 876,663 (Belgium 26 Jan 1960)

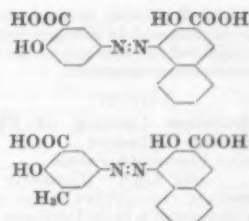
Leuco compounds of basic dyes can be used for hair dyeing, alone or mixed with permanent-waving liquids containing reducing agents. C.O.C.

Temporary Hair-colouring Composition

John H. Breck

USP 2,960,443 (11 Oct 1956)

A mixture of alcoholic solutions of Potachrome Azure Blue BR 200% concentrate (C.I. Mordant Blue 1) and du Pont Dark Brown No. 173 (a mixture of



and

and an aqueous solution of an aluminium salt, is stable and free from precipitate. It is used for colouring hair so that the hair remains coloured until it is washed. C.O.C.

Dyeing Cellulose Triacetate with 1 : 2 Metal-complex Unsulphonated Monoazo and Monoazomethine Dyes

BrC

BP 866,329 (5 Sept 1956)

Filaments of cellulose triacetate obtained by extruding a solution of triacetate in a water-miscible solvent, e.g. acetic acid, into a liquid water-miscible coagulant, e.g. aqueous acetic acid, have, while still wet and swollen after being washed with water to remove all solvent, good substantivity for the 1 : 2 metal complexes of unsulphonated monoazo or monoazomethine dyes in which the azo or azomethine group links two C atoms, each of which is directly linked to a C atom directly linked to OH. Thus wet cellulose triacetate tow is readily dyed fast to washing and to cross-dyeing with Cibalan Blue BL (C.I. Acid Blue 168) by padding it with an aqueous solution of the dye and then steaming for 15 min at 100°C. C.O.C.

Dyeing or Printing Materials containing Polyester Fibres

BASF

BP 875,055 (Germany 25 Mar 1959)

Disperse or vat dyes are readily applied by adding to the dye liquor or printing paste a compound of formula $NC-R-N(X)Y$ (R = alkylene of 1-3 C; X = H or -RCN; Y = N, Alk of 1-4 C, acyl radical of an aliphatic monocarboxylic acid of 1-4 C, or $-CO(CH_2)_n-CO-NX-R-CN$ (n = 0 or 1)), e.g. 2-formamylaminopropionitrile. C.O.C.

Mass-coloured Polypropylene

Montecatini Società Generale per l'Industria Mineraria e Chimica

BP 875,133 (Italy 15 Nov 1956)

A dye stable at the m.p. of the polymer and which has no degrading action on it, e.g. Durindone Scarlet YP (C.I. Vat Red 45), is incorporated into the polymer melt or solution before extrusion. C.O.C.

Auxiliaries for Prevention of Gas-fume Fading of Dyed Cellulose Acetate Fibres (III p. 563)

Procynyl Dyes—New Reactive Dyes for Nylon (IV p. 565)

Azo Disperse Dyes for Protein Fibres (IV p. 570)

Triazines for Azoic Blacks and Dark Blues on Polyester Fibres (IV p. 572)

Anthraquinone Disperse Dyes for Protein Fibres (IV p. 573)

Effect of Sulphuric Acid on Wool. V—Investigation of Dyeing Faults caused by Carbonising (VII p. 579)

Effect of Formaldehyde on Azo Dyes containing Amino Groups in Crease-resist Finishing with Urea-Formaldehyde Resins (X p. 585)

Wash-resistant Effects on Cellulose Textiles (X p. 587)

Light Absorption of Dyes. IV—Relation of Transmission and Reflection of Light by Diffusing Materials to Concentration of Colour (XIV p. 594)

IX—PRINTING

New Photographic Processes. I—The Arylamine-Carbon Tetrabromide System giving Print-out Dye Images

R. H. Sprague, H. L. Fichter, and E. Wainer

Phot. Sci. Eng., 5 (1961) 98-103

Chem. Abs., 55 (21 Aug 1961) 16233

A print-out dye image is formed in a mixture of a polyhalogen compound and an arylamine by irradiation in the near ultraviolet. Reaction of diphenylamine with $CBBr_4$, induced by ultraviolet irradiation, produced a dye with a sharp absorption maximum at 558 m μ in methanol. The dye was Opal Blue SS (C.I. 42760). A free-radical

chain mechanism of formation is suggested. Data are given for 14 other amines and 14 halogen compounds. The images can be stabilised by heating to drive off volatile sensitiser or by solvent extraction. C.O.C.

PATENTS

Applying a Seamless Coating of Photosensitive Material to Printing Cylinders

Dr. Bekk & Kaulen Chemische Fabrik

BP 876,827 (Germany 17 Sept 1958)

During immersion of the cylinder in the coating liquor, and during its withdrawal, a forced draught of filtered air is blown against the surface of the cylinder above the surface of the liquor. This screens the liquor against penetration of dust during lowering and raising of the cylinders, so that no dust can attach itself to or mingle with the coating. C.O.C.

Printing and Stencilling on Polyethylene

Diamond Alkali Co.

USP 2,961,346 (26 May 1958)

A composition consisting principally of a chlorinated paraffin and a small amount of at least one of the following: film-forming agent, plasticiser, "slip" agent and dryer, together with an oil-soluble colorant, all dispersed in a solvent, is readily applied to a polyethylene surface which has not been given any preliminary coating. C.O.C.

Colour Photography

Kodak

BP 874,078 (U.S.A. 8 Mar 1957)

In a reversal process, presence in the developer for the negative images of an ionic polyethylene glycol of mol. wt. < 600 gives increase in developability of at least one of the latent images, which gives effectively more speed judged by the resulting colour positive images.

BP 874,079 (U.S.A. 8 Mar 1957)

A polyethylene glycol of mol. wt. < 400 but preferably 1500–4500, or a non-ionic derivative thereof, is used. C.O.C.

Colour Photography without Use of Silver Salts

DuP

BP 875,248 (U.S.A. 18 July 1958)

A layer comprising (a) a colourless addition-polymerisable ethylenically unsaturated monomer having at least one ethylenic end group and containing a dye nucleus capable of forming an azo, quinoneimine or azomethine dye on reacting with a dye-forming component, (b) a polymerisation catalyst that is activated by actinic light and, if desired, (c) a binder for these ingredients, is exposed to light in the desired areas until substantial polymerisation occurs. Unexposed and unpolymerised material is then removed, and unexposed and polymerised image material portions of the layer are treated with a dye-forming component to produce a coloured image. The last two steps may be carried out simultaneously or in either order. C.O.C.

Additive Colour Photography

International Polaroid Corp. BP 874,990 (25 Nov 1959)

Describes a combination of the two existing types of processing. C.O.C.

Three-colour Xerography

Haloid Xerox

USP 2,962,374–5 (2 May 1956)

Developing Images and Designs on Metal Surfaces

Modern Engineering & Machine Co.

USP 2,962,364 (15 Aug 1958)

The metal sheet, e.g. aluminium, is cleansed and then treated with cold 5–15% aq. HCl saturated with salt for 30–90 s. It is coated with a light-sensitive material at 65–90°F, exposed through a negative, developed, and dyed to make the image visible. Any areas not to be etched are then coated with a resist, e.g. asphalt, and the sheet is then treated with aqueous iron perchloride (40°Bé) containing 10–15 oz HCl per gal. Etching is carried on for a short period in the solution and the sheet briefly exposed to the air, which causes the reaction to become very violent, the sheet being finally immersed in water when the required degree of etching is reached. This yields a frosted background comparable to an embossed background. C.O.C.

Colour-coupling Developers (IV p. 569)

Azo Disperse Dyes for Protein Fibres (IV p. 570)

Improved Solubility of Diazonium Double Salts (IV p. 572)

Stable Azotic Composition Solutions (IV p. 572)

Reactone Dyes in Dyeing and Printing (VIII p. 580)

Wet Processing of Textiles in a Fluidised Bed (VIII p. 583)

Vat Dyeing or Printing Blends of Cellulose Fibres and Fibres of Cellulose Esters of Low Hydroxyl Content (VIII p. 583)

Dyeing or Printing Materials containing Polyester Fibres (VIII p. 583)

X—SIZING AND FINISHING

Drying

P. Y. McCormick

Ind. Eng. Chem., 53 (July 1961) 583–585

A unit operations review, covering the final quarter of 1959 and the whole of 1960, devoted to drying plant. Of particular interest are the description and economics of an atomiser for the spray drying of heavy pastes of inorganic pigments, and investigations into air flow, heat- and mass-transfer problems in production and model spray dryers. Recent research in the drying of wool in bobbin and loose-stock form, the effect of wetting and drying on wool fibre strength, water-retention capacity of cotton fabrics, and methods employed for drying polyvinyl chloride are mentioned briefly. 59 References. F.J.

Antistatic Finishes

K. W. Mieszkis *B.N.S. Outlook*, No. 10 (Spring 1961) 8–15

A general review of the mechanism of operation of antistatic agents, their application to yarns, and the testing of antistatic finishes. M.T.

Review of the Chemistry of Thermosetting Resins and Related Compounds and their Application to Textiles

A. R. Smith

J.S.D.C., 77 (Sept 1961) 416–434

Cross-linking of Cellulose and its Derivatives

II—Preparation of some New Amino-derivatives of D-Glucose and of D-Galactose

W. M. Corbett

J.C.S., (July 1961) 2926–2930

In Part I (cf. *J.S.D.C.*, this issue, p. 543) the addition of amines to unsatd. cellulose esters is outlined as a useful means of cross-linking cellulose and its deriv. For this study the use of amino-deriv. of monosaccharides is essential, and has necessitated the preparations now reported. With methanolic ammonia, 6-deoxy-6-iododi-O-isopropylidene-D-galactose gives mainly 6-deoxydi-O-isopropylidene-6-methylamino-D-galactose, whereas aq. ammonia gives the t-amine with 3 sugar residues, and ammonia in dioxan gives the latter and the s-amine. 2-Aminoethyl ethers of di-O-isopropylidene-D-glucose and -D-galactose are prepared from the corresponding methoxycarbonylmethyl ethers. These cpd. are converted into the amides, which are then reduced to the amines.

III—Addition of Amines to Crotonoyl Esters

W. M. Corbett and J. E. McKay

Ibid., 2930–2935

The addition of some primary aliphatic amines to ethyl crotonate and to 1,2:5,6-di-O-isopropylidene-D-glucose-3-crotonate, and of this sugar ester to amino-deriv. of simple sugars, has been investigated to determine the validity of the application of the work of Stadnikow (*Ber.*, 44 (1911) 52) and Marsh (*Monatsh.*, 60 (1932) 50) to carbohydrate deriv., and to establish, for analytical purposes, the structure of the cross-links formed between mol. H.H.H.

Self-smoothing Fabrics: Chlorine Retention Damage

J. T. Marsh

Text. Manuf., 87 (July 1961) 283–288

The author reviews the history of "chlorine-retention damage" and methods of minimising it. Test methods are described and the use of resin buffers is considered.

W.G.C.

Self-smoothing Fabrics: Use of Formaldehyde

J. T. Marsh

Text. Manuf., 87 (Aug 1961) 327–330

There are two types of process for the treatment of cotton with acidified formaldehyde solutions at room temperature. In the wet-recovery processes the fibres are stabilised in a distended state which is potentially high swelling, whilst in the dry-recovery process the swelling of the cotton is reduced by the addition of acetic acid to the acid-formalin mixture, e.g. a solution containing 70% water gives very good wet recovery and little or no dry

recovery in a fabric with only 0.5% combined formaldehyde, whereas an equivalent wet recovery, accompanied by good dry recovery, obtained from the dry-recovery process with 17% of water requires a formaldehyde content of 0.8–1.0%; consequently the strength is lower.

W.G.C.

Effect of Additives on Reaction between Cellulose and Formaldehyde

W. J. Roff *J. Textile Inst.*, 52 (June 1961) T301

A preliminary note. The addition of small proportions of urea to an acid solution of formaldehyde has been found to have a very marked effect on the retention of formaldehyde in the conventional impregnating, drying, and baking process.

P.G.M.

Crease Recovery

J. T. Marsh *J.S.D.C.*, 77 (Oct 1961) 496–497

Use of Alkyl Keten Dimer as Assistant in Resin-treatment of Cellulosic Fabrics

I. Imai, K. Takahashi, S. Nishikawa, and K. Funayama
J. Soc. Textile Cellulose Ind. Japan, 17 (June 1961) 537–546

Application of softening agents to resin-treated fabrics results in an improvement in the physical properties, and the use of alkyl keten dimers in this role has been studied. The effects of variation in alkyl chain length and concentration of the alkyl keten dimer in the resin-treatment bath on the physical properties of the fabric and the nitrogen content of the resin-treated fabric were investigated. Addition of alkyl keten dimer improved the physical properties of the resin-treated fabric; it also greatly increased the crease recovery (compared to resin treatment alone) owing to the lubricating action. It also improves the water repellency of the fabric when the number of carbon atoms in the alkyl chain exceeds 10, and the effect is reduced only slightly on repeated washing. From determinations of the nitrogen content the following conclusions have been drawn—(1) On treatment of cotton with Sumitex Resin 901 (dimethylol ethylene urea), addition of alkyl keten dimers has no effect on the nitrogen content, i.e. it does not interfere with the cross-linking reaction between the resin and the hydroxyl groups of cellulose. (2) In the treatment of viscose rayon staple with Sumitex Cream 450 (monomethylolurea) the use of alkyl keten dimer results in a decrease in nitrogen content, i.e. it interferes with the deposition of polymerised resin in the micellar interstices of the cellulose. This does not cause any deterioration in crease recovery.

I.G.L.

Effect of Formaldehyde on Azo Dyes containing Amino Groups in Crease-resist Finishing with Urea-Formaldehyde Resins

H. Braun

Textil Praxis, 16 (July 1961) 728–733

A description is given of crease-resist finishing with urea-formaldehyde resins, and of the action of formaldehyde on 14 disazo dyes of the Congo Red type. Possible reactions of free amino groups when present as in Benzo Bordeaux B (C.I. 22155) and Benzo Orange Brown D3G (C.I. 30045), and the reaction mechanism of aromatic amines with formaldehyde in acid media, are discussed.

9 References.

H.H.H.

II

Ibid., 16 (Aug 1961) 792–797
Studies are reported of the reaction between HCHO and *p*-nitroaniline, *p*-aminoazobenzene, *N*-methyl-*p*-aminoazobenzene, and sulphanilic acid → aniline. Dyes containing amino groups may have their substantivity reduced by reaction with HCHO, and methylamines may be split off from the resultant cpd.

J.W.D.

Graft Polymerisation of Acrylamide on to Cotton Fabric for Further Treatments

H. Kamogawa and T. Sekiya

Text. Research J., 31 (July 1961) 585–591

Graft polymerisation of acrylamide on to cotton using ceric ammonium nitrate as catalyst proceeded comparatively quickly, even in the cold. The optimum pH for obtaining maximum addition of polyacrylamide was 1.4–1.6 at 25°C. Much of the polyacrylamide produced in and on the fibre may possibly be grafted to the cellulose molecule, since values for apparent polyacrylamide content nearly similar to those for original fabrics were obtained for samples recovered upon acidification of their

cuprammonium solutions. Of all aftertreatments investigated, acid cure of methylolated polyacrylamide-grafted fibres alone or in conjunction with adipamide gave the best fastness to acid extraction and to alkaline washing, presumably because of formation of a durable methylene-bisamide linkage between graft chains. HCHO-cure of the polyacrylamide-grafted cotton, as well as of the original cotton, also yielded excellent fastness to acid extraction. However, this finish gave fabric poor strength compared with the former, presumably because of formation of short methylene cross-linkages between cellulose molecules by free HCHO. A durable crease-recovery finish, in the strict sense of having the same life as the substrate, has not yet been achieved, but it is possible that graft polymerisation in combination with or followed by cross-linking may provide a means of obtaining such a finish.

C.O.C.

Study of NN-Ethylene Higher Alkyl Urethanes I—Synthesis, and Water-repellent Finishing of Cellulose

Y. Iwakura and Y. Tezuka
J. Soc. Textile Cellulose Ind. Japan, 17 (July 1961) 681–687

Stearyl, cetyl, myristyl, and lauryl chloroformates were prepared by the action of phosgene on the corresponding alcohol in benzene, and converted to NN-ethylene higher alkyl urethanes by condensation with ethylenimine in benzene using triethylamine as acid acceptor. Viscose rayon fabric treated with NN-ethylene stearyl urethane exhibited the best water repellency.

II—Preparation in Water and Finishing

Y. Tezuka and Y. Iwakura *Ibid.*, 687–693
The NN-ethylene higher alkyl urethanes were prepared in water (using soda ash or $(\text{NH}_4)_2\text{CO}_3$ as acid binder). NN-Ethylene stearyl urethane prepared in this way imparts a durable and wash-fast water-repellency to rayon fabric (untreated or treated with urea- or melamine-formaldehyde resin).

III—Test Production and Industrial Finishing Test

Y. Tezuka *Ibid.*, 693–699
Viscose rayon fabric was treated on a large scale with NN-ethylene stearyl urethane prepared by the heat dispersion method. The fabric exhibited durable water repellency and, in addition, improved flexural abrasion resistance, tensile strength, and tear strength.

Recent Developments in Textile Processing

B. C. M. Dorset *Text. Manuf.*, 87 (June 1961) 243–248
A general review of shrinkproofing of wool, developments in oil- and water-repellent finishes, flameproofing, and the modification of wool to improve its properties.

M.T.

Theory of the Shrink-resisting of Wool

II—Chemical Modification of the Fibre Surface and its Effect on Felting Shrinkage, Friction, and Microscopic Appearance

J. H. Bradbury

Text. Research J., 31 (Aug 1961) 735–743

Wool can have its felting power reduced by a variety of chemical and physical surface treatments. The chemical composition of the surface of the treated fibre and its topography (as observed microscopically) vary according to the type of surface treatment. There is good correlation between the amount of surface modification and the change in frictional coefficients. The latter seems to be the basic cause of reduction in felting power. This theory is adequate for most surface treatments, but not for treatment with aqueous permanganate followed by bisulphite, or for extraction with ethanol.

III—Determination of the Force on a Fibre during Felting

J. H. Bradbury and H. Groll *Ibid.*, 743–745

Describes a method of assessing the maximum force exerted on a single fibre during felting of fabric or top.

C.O.C.

Development of a New Shrink-resist Finish for Wool

A. N. Davidson *J.S.D.C.*, 77 (Sept 1961) 409–415

Shrinkproofing Wool with Reducing Agents

A. J. Farnworth *J.S.D.C.*, 77 (Oct 1961) 483–488

Fibre-surface Contaminants on Wool and their Effect on Shrinkproofing

A. J. Farnworth *J.S.D.C.*, 77 (Oct 1961) 489–495

Wool Fabric Stabilisation by Interfacial Polymerisation. I—Polyamides

R. E. Whitfield, L. A. Miller, and W. L. Wasley

Text. Research J., 31 (Aug 1961) 704-712

Wool fabrics are made shrink-resistant by using interfacial polymerisation reactions to form polyamides on the fibre surface. The treated fabrics are dimensionally stable during repeatedly accelerated washings, and are unaffected in handle, flexural rigidity, breaking strength, elongation, and chemical resistance, and are also improved in crease-recovery and smoothness after tumble drying. A variety of polyamides may be used, but best results are obtained with polyhexamethylene sebacamide. The fabric is padded first with an aqueous diamine and then with a water-immiscible solution of a diacid chloride. Polymerisation is extremely rapid, heating and curing being unnecessary. Finally the fabric is washed and dried.

C.O.C.

Setting of Animal Fibres in Solutions of Thiourea Dioxide

L. S. Bajpai and C. S. Whewell

J.S.D.C., 77 (Aug 1961) 350-351

Isocyanate Modification of Wool in Dimethyl Sulphoxide

N. H. Koenig *Text. Research J.*, 31 (July 1961) 592-596

Dry wool reacts with aliphatic and aromatic monoisocyanates in hot dimethyl sulphoxide or pyridine but not in other organic liquids. The action is very rapid, the wool taking up 30% of its weight of phenyl isocyanate in 10 min at 105°C. Wool cloth treated with phenyl isocyanate has good mechanical strength and crease recovery, but colour, handle, and flexibility are more or less impaired. Fabrics modified by various isocyanates are generally more resistant to acids, alkalis and oxidising agents, and are dimensionally stable to laundering.

C.O.C.

Influence of Changes in Moisture Content on the Crease Recovery of Fabrics

I

A. J. Farnworth and J. Lindberg

Text. Research J., 31 (Aug 1961) 687-694

Wool, wool-blend, and cotton fabrics show two-stage recovery from wet creasing. Following a normal recovery there occurs a second-stage rapid recovery at a regain corresponding to the point at which the rate-of-drying curve ceases to be linear, i.e. when drying changes from surface evaporation to loss of water from inside the fibre. In evaluating crease recovery from wet creasing, the recovery angle should be measured only after the fabric is in equilibrium with the atmosphere. Crease recovery after wet creasing is independent of the rate of drying. The above fabrics, after reaching equilibrium crease recovery after wet creasing, show additional progressive, step-wise recovery when repeatedly rewetted and redried. Fabrics having very poor normal recovery from wet creasing can ultimately show excellent recovery after several such cycles. The same behaviour recurs with alternate steaming and drying cycles. The effect results only from alternating increase and decrease in regain, since prolonged immersion in water, prolonged drying, or rewetting without intermediate drying do not give additional crease recovery. Probably the progressive recovery is caused by some of the fibre and yarn interaction and frictional forces responsible for maintaining the fabric deformation being overcome by the small forces generated by the swelling-deswelling process.

II

J. Lindberg

Ibid., 695-696

When a dry-creased wool fabric is rewetted, complete crease recovery occurs, but when a wet-creased and dried wool fabric is rewetted there is no recovery at all, but progressive recovery upon alternate drying and wetting does occur. Dry-creased cotton fabric does not completely recover on wetting, but both dry-creased and wet-creased cotton fabric show progressive recovery on alternate drying and wetting. Mathematical consideration, using the rheological models proposed by Lindberg and by Olofsson (*Text. Research J.*, 31 (1961) 99 and *J. Textile Inst.*, 52 (1961) T272, respectively), has established that the theory of frictional restraint is compatible with the progressive recovery on alternate drying and wetting and gives a far better explanation than the theory of permanent fibre deformation.

C.O.C.

Effect of Silicone Finishing on Tensile Strength of Wool Fabrics

W. Heydeck *Melliand Textilber.*, 42 (July 1961) 803-805

Silicone-treated wool uniform fabrics were tested, at various stages of the treatment, for changes of tensile strength. The suspected deleterious influence of the treatment was not borne out by the results obtained.

P.B.S.

New Methods of Processing Raised Wool-containing Fabrics

N. V. Sevast'yanov

Tekstil. prom., No. 3 (Mar 1961) 42-43

A detailed method of raising and finishing circular-knitted fabrics is given. After dyeing, the excess of moisture is removed by suction slot or centrifugal hydro-extraction. This is followed by dressing in width and festoon-drying. Fabric is then cut open and raised (two passes). For best results, fabrics containing 50% wool should have a moisture content of 10%. Raising is followed by cropping and dressing in width.

L.S.L.

Use of Chromium Salts to Improve the Resistance of Wool to Moisture and Microbiological Action

G. Nitschke *Melliand Textilber.*, 42 (July 1961) 818-821

A short review of previous work is given with a table of the various treatments and their effects. The use of chromic fluoride or chromic anhydride in the fibre treatment gave a better result than the simpler treatments and corresponded to the effect of potassium chromate-copper sulphate treatment. Combined chroming and moth-proofing treatment gave a better result than that obtained by the treatments applied separately.

P.B.S.

Permanent Pleating of Polyester/Wool Mixtures

H. Görlach *Chemiefasern*, 11 (July 1961) 472-477

Problems connected with permanent pleating (i.e. stability to washing) of polyester-wool mixtures are considered with fibre properties in mind. Three common methods of pleating are discussed—use of a steam oven at either 100°C or 130-150°C (with superheated steam) or pleating machine (contact temperature 150-160°C)—and the advantages and disadvantages of each cited. Pleating by ironing or steam pressing is not recommended. A means of testing pleating stability is described, and details of temperature measurements in steaming ovens are given. The use of tulle as an elastic lining between fabric and mould is reported to minimise crimping under certain conditions, but results in some reduction in sharpness of pleating. The use of moulds used previously for resin-treated cotton fabrics is not recommended, as changes in colour are likely to occur owing to the presence of formaldehyde residues. Moulds contaminated with formaldehyde should be steamed twice before further use. Yellowing of white fabrics may occur. It is recommended that the sewing material should be subjected to the pleating process with the fabric to minimise the risk of any differences in colour being apparent in the finished garments.

I.G.L.

Setting Nylon Fabrics by Radiant Heat

L. S. Laskowski

B.N.S. Outlook, No. 10 (Spring 1961) 29-35

A discussion of methods of setting is followed by a more detailed account of the infrared setting of nylon. Graphs are given showing the effect of setting temperature, exposure time, tension during setting, and pre-heating temperature on fabric stability, and the effect of setting temperature on whiteness. The effect of setting on fabric properties is discussed.

M.T.

PATENTS

Sighting Colours for Natural and Synthetic Polymer Fibres

E. A. Murray *USP* 2,959,461 (18 Sept 1956)

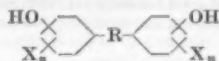
The coloured water-soluble metal chelates of nitroso dyes, when combined with poly-N-vinyl lactams, are far more fugitive than any sighting colours hitherto known. Thus an aqueous solution containing 0.5% Naphthol Green B (C.I. 10020) and 3% poly-N-vinylpyrrolidone, K-30 (General Aniline) is readily removed from wool, acrylic, polyester, nylon, viscose rayon and cellulose acetate fibres, even after steaming for 1 h.

C.O.C.

Rendering Permeable Materials Bacteriostatic

Armour & Co. BP 874,027 (U.S.A. 12 June 1958)

Textiles, leather, paper, and the like are treated with a detergent, a compound of formula



(X = Cl or Br; n = 1, 2 or 3; R = S or alkylene of 1-4 C), and a polyhalogenocarbonyl polyhalogenosalicylanilide. Thus cotton cloth washed in a 0.2% solution of a commercial detergent composition (made of a non-ionic detergent, Na silicate, Na and K pyrophosphates), 3,4,4'-trichlorocarbonyl, and 5-chlorosalicylic acid-3', 4'-dichloroanilide, had good bacteriostatic properties.

C.O.C.

Wash-resistant Effects on Cellulose Textiles

ICI BP 875,364 (17 Mar 1959)

The textile is impregnated with an aqueous solution containing a material capable of reacting with the cellulose and then, while moist, placed in contact with a pair of electrodes and subjected to an electric current in presence of an electrolyte. Both white and coloured effects can be obtained by this means. Thus cotton cloth is padded with its own weight of an aqueous solution of the dye of Example 1 of BP 209,723 (J.S.D.C., 41 (1925) 336) (20 parts per 1000 water), NaCl (5), and an alkylphenol-ethylene oxide condensate (1). It is then sandwiched between two stainless steel plates connected to a source of 12 v D.C. and the current passed through for 12 s. It is then soaped at the boil. This yields a bright bluish red dyeing fast to washing and light.

C.O.C.

Boarding of Knitted Articles in a Fluidised Bed

Hosiery & Allied Trades Research Assocn.

BP 875,757 (24 Nov 1956)

The articles are drawn into forms and then immersed in a fluidised bed of solid, heat-conducting particles. Apparatus is described in which that part of an article first entering the bed is the first to leave it, thus ensuring a uniform period of treatment for all parts of the article.

C.O.C.

Compressive Shrinking

Clupack

BP 875,526 (U.S.A. 20 Jan 1958)

The web to be treated passes between a blanket which passes partly round a roller. A non-rotating pressure bar having a smooth rounded surface presses the blanket against the web in contact with the roller. Two or more pressure-applying devices along the length of the bar cause the bar to press with uniform pressure across the width of the blanket.

BP 875,528 (U.S.A. 26 Oct 1959)

The pressure-applying device is a rotatably mounted roller having a smooth surface. It guides the blanket to and presses it against the nip-forming member. There are means to supply a lubricant between the blanket and the pressure roller to provide a hydrodynamic lubricating film which eliminates the driving friction, so that movement of the blanket does not cause the roller to rotate.

C.O.C.

Pleating Crease-shedding Cellulose Fabrics by Treating with an Acid Catalyst and Hot-pressing

National Cotton Council of America

USP 2,957,746 (11 Jan 1957)

The bonds between the fibres or between the cellulose molecules and the resin are temporarily broken by application of an acid. The pleat is then formed, and heat and pressure are applied, when the acid acts as a catalyst to re-form the bonds between the fibres or between the cellulose and the resin and so set the fabric in the pleated state. Thus cotton cloth rendered crease-shedding by treatment with a cyclic ethylene urea-formaldehyde resin was moistened with 2% aq. Zn(NO₃)₂ at 120-180°F along the area to be pleated. After the solution had been absorbed, the pleat was formed and hand-pressed with an iron at 300-350°F for 1 min. This yielded a sharp pleat which was not removed when the cloth was washed.

C.O.C.

Pleating Fabrics containing Thermoplastic Fibres

Chemstrand

BP 875,575 (U.S.A. 13 Aug 1958)

Cocking, puckering, rippling, or bubbling during and after pleating of the fabric is prevented by forming the damp fabric into a pleating pattern and extending it to

80-90% of the maximum extendable length of the assembly, adjusting it to the pleat-setting position, wrapping and binding it, heating it at < 200°F until dry, and slowly cooling at > 6 deg. F per min until the interior of the assembly is at > 90°F.

C.O.C.

Shrink-resistant Finishing of Knitted Tubular Fabric

Meyer-Waespi & Cie's Successors

BP 874,916 (Switzerland 24 Jan 1957)

The fabric is laterally stretched and thereby shortened. It is then fed between a pair of endless conveyors guided by a series of rollers. The conveyors hold the fabric so that it cannot move longitudinally relative to the conveyors, but so that the lateral tension can be released by a predetermined amount. A predetermined length of one of the conveyors is displaced normally away from the other conveyor. The fabric is then fixed and pressed.

C.O.C.

Imparting Water-repellency to Wool

E. Robbart

USP 2,961,338 (7 May 1958)

Wool of regain < 10%, preferably 4-7%, is treated with the vapours of an organosilicon halide, neutralised, and rinsed. The water-repellent finish obtained is fast to repeated washings.

C.O.C.

Shrink-resisting Finish for Wool Fabrics

Hercules Powder Co.

USP 2,961,347 (13 Nov 1957)

Application of 0.5-10.0% of a cationic thermosetting polyamide-epichlorohydrin resin in aqueous solution at pH 3-10, drying at 65°C for 5 min, and then baking at 100°C for 5 min gives a good shrink-resisting finish without affecting the handle or colour. The resin is obtained by treating a C₈-C₁₀ satd. aliphatic dicarboxylic acid with a polyalkylene polyamide to produce a water-soluble polyamide containing -NH(C₂H₅NH)₂-COR-CO-units (n and x > 1; R = divalent hydrocarbon radical of the acid), which is then reacted with epichlorohydrin.

C.O.C.

Reducing the Felting Power of Wool by Mechanical Treatment

U.S. Secretary of Agriculture

USP 2,958,910 (23 Mar 1959)

Wet or dry wool fibres are drawn over wires whose sides have minute cutting edges or teeth. The size of these teeth is such that they selectively engage the scale structure of the wool and smooth it by a cutting or abrading action without damaging the fibres. This descaling removes only a small amount of material from the fibres.

C.O.C.

Elastic Coating and Impregnating Compositions Compatible with Daylight Fluorescent Pigments

Switzer Brothers

USP 2,963,382 (23 Dec 1957)

Hexamethylenetetramine dispersed in chlorosulphonated polyethylene yields a composition which is readily cured without further addition. Daylight fluorescent pigments added to such a composition are affected neither by the hexamethylenetetramine nor by any solvent for the chlorosulphonated polyethylene. Curing time is only about one-third that previously required; e.g. 5 min at 150°C is satisfactory. The coatings yielded are flexible, very adherent to flexed substrates, and very resistant to abrasion and to weathering, being, e.g., very suitable for tarpaulins.

C.O.C.

Anchoring the Pile of Needleloom Felt

American Felt Co.

USP 2,959,509 (15 Aug 1955)

A layer of pile-forming material is needle-punched either separately or upon a base fabric. A fusible or thermoplastic powdered resin is sifted into the layer or layers and heated to make it adhesive. The heat is wholly or partly supplied by the frictional heat developed by the needles passing in and out of the layers.

C.O.C.

Forming Graft Polymers on Shaped Polymeric Organic Substrates

DuP

USP 2,956,899 (18 Oct 1950)

The substrate is irradiated in presence of about 10¹⁸ to 5 × 10¹⁸ mol. of oxygen/cm² of substrate surface and then stored, if desired, in contact with the desired monomer, with access to any additional quantity of oxygen. Thus dry nylon and polyethylene terephthalate fabrics were placed in a sealed glass vessel evacuated to 1 mm pressure and then exposed to 125 watt-sec/cm² of 2 meV electrons from a Van de Graaf accelerator. After irradiation, the

vessel and its contents were stored for 24 h at room temperature. It was then evacuated to 0.05 mm pressure and opened to a similarly evacuated system containing degassed liquid methyl acrylate. They were kept in this state in the dark for 15 h. The fabrics were then removed from the vessel and extracted in a Soxhlet for 15 h with acetone to remove excess of monomer and loosely attached surface polymer. The nylon fabric increased in weight by 274% and the polyester fabric by 160%. They had a stiffer, tighter, and drier handle and were much less readily penetrated by water. Unirradiated nylon similarly exposed to methyl acrylate vapour increased by only 0.6%. C.O.C.

Grafted Synthetic Addition Polymers

DuP BP 875,131 (U.S.A. 4 Oct 1956)
A synthetic addition polymer is subjected to high-energy particulate or electromagnetic ionising radiation and treated either simultaneously or while it is still activated with a polymerisable monomer containing reactive non-aromatic carbon-carbon unsaturation, and acid or acid salt groups. Examples of suitable substrates are polyacrylonitrile and polyethylene, suitable monomers being acrylic, methacrylic, itaconic, maleic, and crotonic acids. Thus polyacrylonitrile fabric and polyethylene film irradiated in presence of acrylic acid have greatly improved resistance to hole melting. C.O.C.

Treating a Glass-fibre Fabric to Render it Dyeable and Improve its Handle and Draping Properties

Moretex Chemical Products USP 2,961,344 (1 Feb 1957)
Undegraded and incompletely deacetylated chitin is dissolved in aqueous acid. The glass fibre or fabric are then padded with the solution, dried at 100–220°F and then baked, e.g. at 250–275°F to set the chitin. The product can be readily dyed with direct, acid, and azoic dyes to yield dyeings fast to washing. The handle of the fabric is improved. Softeners, plasticisers, and hardening agents, e.g. HCHO, may be added to the solution to modify the finish obtained and increase its durability and fastness to washing. C.O.C.

Water-soluble Silanes as Sizes for Glass Fibres

Union Carbide Corp. USP 2,965,515 (23 May 1955)
Vinylsiloxanes of formula $R^1Si[(OC_2H_5)_2OR^2]_n$ ($R^1 = \text{vinyl}$; $R^2 = CH_3$ or C_2H_5 ; $n = 2-7$), used in aqueous solution and in presence of a catalyst, are good sizes for glass-fibre fabrics which are tube-laminated with polyesters and the like. Thus glass-fibre cloth was heat-cleaned and then dipped into an aqueous solution of vinyl tri-2-methoxyethoxysilane containing NaOH as catalyst, air-dried, and baked for 20 min at 125°C. When laminated with a polyester resin a product was obtained which was resistant to boiling water for 2 h. C.O.C.

Bonded-fibre Fabrics having an Embossed Appearance

Chicopee Manufacturing Corp. USP 2,958,608 (18 Apr 1958)
Fabrics of greater bulk and soft handle are obtained by applying the adhesive in spaced areas or lines and then mechanically treating the bonded fabrics so as to keep the binder areas smooth and unchanged while puffing out or distending the non-bonded portions out of the plane of the basic fabric. C.O.C.

Laminated Decorative Sheets

Th. Goldschmidt USP 2,964,426 (20 Nov 1958)
A sheet of decorative material, e.g. cloth or paper, is impregnated successively with two different types of melamine-formaldehyde resin. The resulting impregnated sheet is particularly suitable for pressing to cause it to adhere to a foundation material because it has a composite resin film, the resins in which have different flowability and hardening properties. The portion of the resin film to be next to the foundation material should have the lower flowability and the more rapid hardening properties. C.O.C.

Decorative Foam Surface Coverings

Congleum-Nairn USP 2,961,332 (21 July 1959)
A backing material is embossed or otherwise has depressions created in it. Distinct portions of a design are

then printed on it with two thermoplastic resinous compositions containing different amounts of foaming agent. It is then heated to fuse and foam the resinous composition. This results in an overall three-dimensional appearance caused by the textured backing and different surface levels caused by the varying amounts of foaming agent in the compositions. C.O.C.

Compounds for Imparting Crease Resistance—the Urons (III p. 563)
Metallic, Reflective Coating on Glass Beads for Incorporation in Coating Compositions (V p. 576)
Setting of Wool Fabrics—a Theoretical Study (VI p. 577)
Relaxation Shrinkage of Wool Fabrics—Its Release with Regain and Time (VI p. 577)
Wet Processing of Textiles in a Fluidised Bed (VIII p. 583)
Dyeing and, if desired, Simultaneous Permanent Waving of Hair (VIII p. 583)
Imparting Bacteria-repellency to Polyvinyl Chloride (XIII p. 592)
Behaviour of Antistatic-finished Textiles in Wearing and Washing Tests—III (XIV p. 595)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Control of Specific Homogeneity of Cellulose by Varying Temperature of Treatment with Dilute Nitric Acid

B. A. Zakharov, V. I. Ivanov, A. L. Maltseva, and G. A. Krylova *Izvest. Akad. Nauk S.S.S.R., otdel. khim. nauk*, (No. 5) (May 1961) 926–927
The number of prevailing molecules determines the degree of homogeneity of cellulose and their average molecular weight determines the specificity of homogeneity. It has been found that the degree of homogenisation of cellulose by dilute nitric acid may be varied by varying the concentration of the acid and the time of the treatment, while its specificity may be controlled by temperature and time of that treatment. I.G.

Action of the Braids Copper-number Reagent on Hydrocelluloses

R. L. Colbran and G. F. Davidson *J. Textile Inst.*, 52 (June 1961) T291–T297
It is shown that the last stages of the reaction between a hydrocellulose and the copper-number reagent are extremely slow, and that the residue from the standard method of determination still possesses appreciable reducing power. The reactions involved lead to the production of carboxyl groups, and about 22 atoms of copper are reduced per carboxyl group formed. The theoretical implications of these observations are discussed. P.G.M.

Infrared Spectrographic Study of Cellulose Acetate

P. Bouriot *Bull. Inst. Text. France*, (94) (May–June 1961) 7–19
Positions of peaks due to acetate groups and hydroxyl groups were investigated for films of cellulose acetate (DS 0.74) (I), secondary acetate (II), and triacetate (III). The acetate C=O peak, which is at 1752 cm⁻¹ in III, shifts to lower frequencies as the proportion of free OH groups increases, owing to hydrogen-bond formation. Conversely, drying of I and II shifts this peak to a higher frequency. The position of the O–H peak at 3480 cm⁻¹ in II confirms the existence of hydrogen-bond interactions with carbonyl groups. In I this peak is at 3446 cm⁻¹, intermediate between the positions for II and regenerated cellulose. Investigation of highly stretched sheets of II, using radiation polarised normal and parallel to the direction of stretch, shows that the acetate group lies in a plane normal to the direction of the chains, whilst the O–H bonds are approximately parallel to this direction. J.C.F.

PATENTS

Pulp of Increased Freeness

American Cyanamid BP 873,560 (U.S.A. 6 Oct 1958)
Freeness is increased by the addition of 0.001–0.01% (based on the dry wt. of fibres) of an anionic vinyl polymer of mol. wt. > 10⁵ to the pulp suspension after sizing with alum and resin below pH 7. R.A.

Paper containing Synthetic-polymer Fibres

Portals BP 873,700 (24 July 1958)

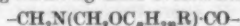
A nylon binder, e.g. methylmethoxynylon, is added to the pulp-synthetic fibre stock, or to the finished sheet, and the paper so produced is treated with a cross-linking agent, e.g. an aldehyde, di- or tri-basic acid, or a urea derivative, in presence of sulphuric acid as catalyst. R.A.

Wet-felted Fibrous Sheet Material containing Polyamide

Société de la Viscose Suisse

BP 874,249 (Switzerland 14 Apr 1958)

Addition to a paper-making slurry which may or may not contain polyamide fibres of an *N*-alkoxypolyamide resin containing recurring units of formula



(*N* = amide atom of a polyamide chain, *n* = 1-6; R = H or OH) and, as catalyst, maleic, nitric, or boric acid or NH_4Cl , yields a paper of excellent strength. The porosity of the paper can be varied within wide limits without affecting the strength. C.O.C.

Paper Sized with Aqueous Emulsions of Keten Dimers

Hercules Powder Co.

USP 2,961,367 (27 Feb 1957)

Improved retention of the dimer by the fibres is obtained by adding to the aqueous pulp suspensions 0.001-0.2% (on wt. of pulp) of a polymer of an alkyleneimine of < 5 C. This enables the same sizing effect to be produced with less dimer and also a small dimer content in the discarded white water. C.O.C.

Surface-sizing with Hydrous Zirconium Compounds of Fibrous Sheets containing Voids

Reheis Co. and Adelphi Research & Manufacturing Co.

USP 2,964,403 (1 Sept 1955)

Paper or the like is coated with sufficient of a dispersion of a hydrous zirconium compound, e.g. $\text{Zr}(\text{OH})_4$, in an aqueous dispersion of an adhesive, to fill voids at the surface and yet leave the fibres exposed between the voids after drying. C.O.C.

Drum-finished Coated Paper

S. D. Warren Co.

USP 2,950,989 (17 Mar 1958)

Rate of production is considerably increased by treating the coated surface with steam close to where it contacts the drum, and/or releasing steam into the entrance of the rib between the coated paper and the finishing surface. It also prevents entrapment of air in the nip, even if no liquid pool is present in the nip entrance, and removes any air in the paper or any entrained or dissolved in the coating. C.O.C.

Improving the Strength of Paper by Treatment with 1,3,5-Triazine Compounds

ICI

BP 875,229 (20 Dec 1957)

The strength, especially the wet strength, of paper is increased by treating it in presence of an acid-binding agent with an aqueous solution or suspension of a compound containing at least one 1,3,5-triazine nucleus substituted in the 2-position by Cl, Br, or a sulfo group, in the 4-position by Cl, Br, sulfo, phenyloxy, or phenylthio group, and in the 6-position by phenyloxy, phenylthio, phenylamino, or naphthylamino group. The phenyloxy, phenylthio, phenylamino, or naphthylamino group may contain a methylhydroxy, alkoxy, amino, subst. amino, carboxy or sulfo group or Cl as a ring substituent. Suitable compounds include 2,4-dichloro-6(3'-sulphoanilino)-1,3,5-triazine and 2,4-dichloro-6-anilino-1,3,5-triazine. C.O.C.

Coated Paper

Consolidated Water Power and Paper Co.

BP 873,682 (U.S.A. 28 Feb 1958)

Paper is first prime-coated with a composition of high adhesive content and, after drying, trailing-blade coated with a second coating containing mineral pigment and a smaller amount of adhesive. The product is a high-gloss book, label, or box-cover paper of improved smoothness, opacity, brightness, and ink hold-out. R.A.

Coating Paper with Polystyrene

Monsanto Chemical Co.

USP 2,961,419 (7 Nov 1956)

Tack-free coatings are obtained by use of an aqueous dispersion containing a polystyrene (100 parts) and an

organic solvent (10-75) of b.p. 175-325°F, and heating the coated paper to < 50 deg F above the b.p. of the solvent. C.O.C.

Mould-inhibiting Paper

Mosinee Paper Mills Co.

BP 876,069 (U.S.A. 30 Aug 1961)

Paper is given good mould-inhibiting properties by coating it with the reaction product of biphenyl, a substituted monohydroxy phenol, and a rosin amine. C.O.C.

Coating Foils with Aqueous Dispersions or Emulsions of Synthetic Polymers

J. P. Bemberg

BP 876,139 (Germany 6 Feb 1959)

Smooth and clear films are obtained, particularly on cellulose foils, by placing the self-supporting foil under tension, initially by shrinkage, and then leading it in a completely smooth condition over the coating roller. It is then briefly shock-dried at high temperature by infrared radiation, drying being completed in the conventional manner. During all these processes, constant tension is maintained in the foils. C.O.C.

Coating Compositions for Metal and Paper (V p. 576)

States of Aggregation of High-molecular Compounds.

VIII—Effect of Temperature on Ramie Fibres and

Cellulose Hydrate Films (VI p. 577)

Laminated Decorative Sheets (X p. 588)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS**Standard Definitions of Terms Relating to Leather. ASTM Designation D.1517-60**

J. Amer. Leather Chem. Assocn.,

56 (July 1961) 366-390

The definitions are subdivided into two groups: Group A comprises terms applicable to the sampling of leathers for determining their chemical and physical properties. Group B includes some of the terms encountered by personnel concerned with specifications for the description of leather quality and for leather procurement. Terms adequately defined by unabridged dictionaries are not included. This is termed "ALCA Method X I"

J.W.D.

Radiolabels and their Applications

J. A. Moss

J. Soc. Leather Trades Chem., 45 (Aug 1961) 321-329

Large radiation sources are used for the sterilisation of medical products and the vulcanisation of rubber. Sources of lower intensity are used in measuring instruments, e.g. liquid-level indicators and thickness gauges. Tracer techniques are briefly discussed; though these have not yet been much used in the leather field they are potentially useful in studies of chrome complexes and the stability of leather, investigations into the penetration of substances into pelt, and the irradiation sterilisation of hides and skins. J.W.D.

Recent Advances in Knowledge of Structure of the Collagen Fibril and of Properties of the Tropocollagen Macromolecule

J. H. Highberger

J. Amer. Leather Chem. Assocn.,

56 (Aug 1961) 422-455

Survey of the present state of knowledge; there are numerous diagrams and electron micrographs. J.W.D.

Nature of the Linkage of Glutamic Acid in Collagen and Elastin

K. T. Joseph and S. M. Bose

Bull. Cent. Leather Res. Inst., Madras, 7 (1960) 97-111

J. Amer. Leather Chem. Assocn., 56 (Apr 1961) 193-194

The stability of collagen has been ascribed to various cross-links, one of which may involve the γ -COOH of glutamic acid residues. γ -COOH links are resistant to tryptic hydrolysis. Purified collagen, alkali-process gelatin, and elastin were partially hydrolysed with trypsin or elastase. The hydrolysates were oxidised with hypobromite, and the resulting succinic acid from glutamic acid not linked through γ -COOH was extracted with ether. The extracts were hydrolysed with HCl to break γ -COOH bonds, and the hydrolysed soln. were oxidised and ether-extracted as before. Succinic acid from glutamic acid

combined through γ -COOH was then determined. The amounts found corresponded to 1.3 mmole/100 g for collagen, but only 0.17 and 0.14 mmole/100 g for gelatin and elastin, respectively. The low value for gelatin indicates that most of the γ -COOH links of collagen are broken by the alkaline-gelatin process. It also appears that γ -COOH groups in collagen are bound to part of the arginine or lysine residues, since only 67% of arginine and 72% of lysine residues of collagen are set free by tryptic hydrolysis. M.T.

Hydrotropic Attack of Surface-active Materials on Hide Substance

H. Loewe and G. Otto *Das Leder*, 11 (1960) 251-256
J. Amer. Leather Chem. Assoc., 56 (Apr 1961) 197-198

Many wetting agents used in tanning damage the hide. In neutral media they swell or dissolve collagen by hydrotropic action, i.e. by their attraction for water. Anionic agents are more hydrotropic than cationic ones. Alkylsulphates lower the shrinkage temp. of hide if of chain length C_{12} - C_{14} . Anionic materials have greatest action on raw hide and chrome-tanned leathers, whereas cationic ones tend to attack vegetable-tanned leather. The protective action of certain surface-active substances against hydrotropic attack on collagen is discussed in detail. M.T.

Sorption of Water Vapour by Hydrophilic High Polymers. V—Sorption Isotherms and Heats of Wetting of Collagen and its Derivatives

E. F. Nekryach *Ukrain. khim. zhur.*, 27 (No. 3) (1961) 330-335

The isotherms of water vapour absorption at 20°C and heats of wetting of crude powdered hide and of hides treated with chromium salt, tannin, or formaldehyde were determined. Water sorption decreases in the following sequence of treatments—chromium salt > formaldehyde > crude hide > tannin. Heats of wetting decrease as follows—crude hide > chromium salt > formaldehyde > tannin. The values of sorption and heats of wetting differ little above moisture contents of 35-40%, with the exception of tannin-treated hide. This exception is explained by changes in the collagen structure. The bonding of water by collagen is accompanied by a decrease in differential entropy. The strength of bonds between various functional groups of collagen and water molecules is deduced from the location and values of entropy minima. The variation of differential enthalpy, depending upon moisture content in various samples of hide, indicates the comparative values of hydration energy of various functional groups in collagen, which form the following sequence



I.G.

Report of Symposium on Retannage of Chrome-tanned Leather

J. Amer. Leather Chem. Assoc., Suppl. No. 7 (1960) 50 pp.
Brief reports on vegetable-extract, chrome, zirconium, and resin retannage, and verbatim account of discussion. M.T.

Report of Symposium on Vegetable Tanning

J. Amer. Leather Chem. Assoc., Suppl. No. 8 (1961) 53 pp.
Brief papers on scientific principles of vegetable tanning and vegetable tanning of sheepskin; upholstery; bag, case and strap; and sole leathers, followed by verbatim report of discussion. M.T.

Tanning and Dyeing of Glove Leather

W. Bruegel *Leath. Trade Rev.*, 135 (1960) 261
J. Soc. Leather Trades Chem., 45 (June 1961) 265

Various processes for producing white gloving leather are discussed and some hints on dyeing are given. Leathers fat-liquored before being dyed have greater rubbing fastness than those fat-liquored after being dyed. M.T.

Treatment of Leather with Fluorocarbon Resin

R. Gourlay *Riv. tech. Industr. Cuir*, 52 (1960) 33
J. Soc. Leather Trades Chem., 45 (June 1961) 265

A fluorocarbon resin is said to give results at least as good as, and sometimes better than, those obtained with two silicone-based preparations. M.T.

Relation between Wearability and Thermal Stability of Leather

G. I. Kutyanin and K. G. Karimov

Doklady Akad. Nauk S.S.S.R., 138 (No. 3) (21 May 1961) 625-627

Shrinkage temperatures and abrasion resistance were determined for a series of leather samples prepared from the same hide, but tanned with different tanning agents, e.g. tannin, formaldehyde, chromium salts. A linear relationship has been found between these two parameters. As the shrinkage temperature of leather depends upon the structure of collagen, the results confirm the assumption that wear of leather is due to the breaking of intramolecular bonds of collagen. I.G.

Improvement of some Properties of Sole Leathers by Impregnation with Polymers

J. R. Kanagy *J. Amer. Leather Chem. Assoc.*, 56 (July 1961) 322-342

Impregnation of leather with organic elastomers and resins will improve the wear of sole leather by at least 50%. Optimum effect is attained if the leather is impregnated in the crust state at the tannery, without addition of inorganic fillers, and is then rolled as in current practice. Results are given which indicate that tests of water absorption and abrasion resistance may be used as a basis for performance standards for impregnated leathers. There are 2 photomicrographs. J.W.D.

PATENTS

Waterproof Leather

Socony Mobil Oil Co. *USP* 2,964,425 (29 Apr 1958)

Treatment, if desired in conjunction with a stuffing grease, with a compound of formula $R-NCH_2-CH_2COOH$ (R = radical of an aliphatic alcohol of 10-22 C), e.g. oleoyl or stearyl sarcosine, renders leather waterproof without rendering it impermeable to water vapour. C.O.C.

Increasing the Resistance of Leather to Water

Boehme Fettchemie

BP 875,711 (Germany 28 June 1957)

Modifications of *BP* 791,633 (*J.S.D.C.*, 74 (1958) 445). Much improved results are obtained if the leather is treated with an aqueous solution or dispersion of a derivative of an inorganic or organic acid of basicity ≤ 3 , which derivative is rendered lipophilic by containing a high-mol. wt. aliphatic, cycloaliphatic, aliphatic-aromatic or aliphatic-cycloaliphatic residue. It is preferable that this derivative shall contain more than one free salt-forming group capable of complex formation with the polyvalent metals used in the parent specification. Examples of a suitable derivative are the NH_4 salt of an acid phosphoric acid dodecyl ester and the monododecyl ester of citric acid. C.O.C.

Reaction between Perfluoro-octanoic Acid and the Fibrous Proteins Keratin and Collagen (VI p. 578)

Leather Dyeing using Acetone as Solvent (VIII p. 583)

Occurrence of Pyrrole-2-carboxylic Acid in Hide Hydrolysates and its Bearing on the Determination of Hydroxyproline (XIV p. 596)

Design of Methods for Testing Fastness of Light Leathers to Rubbing Under Various Conditions (XIV p. 596)

Examination of Rub Test for Determination of Fastness of Leather Finishes (XIV p. 596)

XIII—RUBBER; RESINS; PLASTICS

Infrared Spectra of Polyacrylonitrile Polymerised by γ -irradiation at Low Temperatures

Y. Tsuda

Bull. Chem. Soc. Japan, 34 (July 1961) 1046-1048 (*in English*)

Acrylonitrile was polymerised in bulk in various soln. by γ -radiation at 17, -78.5, and -196°C, and the intensities of the absorption bands at 1675 and 2030 cm^{-1} were found to vary according to the solvent species (e.g. triethylamine and dimethylformamide) and the irradiation temp. From its behaviour, the band at 1675 cm^{-1} is assigned to a conjugated C=N linkage produced by irradiation. The band at 2030 cm^{-1} is due to the ketene-imine linkage. H.H.H.

Polyesters for Surface CoatingsJ. G. Weinmann *Off. Dig. Fed. Soc. Paint Technol.*, 33 (July 1961) 842-854

A review including discussion of formulation variables, methods and problems of application, and comparison of polyester-based finishes with conventional surface coatings. Polyester coating formulations suitable for application to wood and masonry are discussed. J.W.D.

Equilibrium between Crystalline and Amorphous Phases in Polyethylene

R. Chiang and P. J. Flory

J. Amer. Chem. Soc., 83 (5 July 1961) 2857-2862

Crystallisation and melting of fractionated linear polyethylenes (Marlex-50 and a low-pressure polyethylene) have been investigated by dilatometry. Melting of fractions crystallised at the highest feasible temperature (131.3°C) were sharp. The m.p. characterised by a reduction to 0% in crystallinity, for a fraction of mol. wt. 490,000, was determined as 136.5-138.5°C. The m.p. of 138.5°C is the highest yet recorded for polyethylene. Melting of binary mixtures of a polyethylene fraction with α -chloronaphthalene over the temp. range immediately preceding final disappearance of crystallinity has been studied. The binary phase is assumed to comprise amorphous polymer and diluent. Results show agreement with the thermodynamic expression relating temperature to liquid composition up to a degree of crystallinity as high as 50%. It is concluded that attainment of phase equilibrium in polymer systems is not restricted to very low degrees of crystallinity. F.J.

Infrared Spectra of some Deuterated Crystalline Polystyrenes. III—Characteristic Bands of Isotactic Polymer from Styrene- d_5

M. Kobayashi

Bull. Chem. Soc. Japan, 34 (July 1961) 1045-1046 (in English)

The preparation of the monomer $C_6D_5CH=CH_2$ is briefly outlined. Polymerisation of it in *n*-heptane (Ziegler catalyst) affords isotactic polystyrene- d_5 and bulk polymerisation (B.P.O. catalyst) gives the atactic polymer. The infrared spectra of these cryst. deuterated polymers show that the polarisation properties of the characteristic bands coincide with those of isotactic polystyrene, and indicate that the C-H deformations of the phenyl group have little effect, especially in the region 1450-1180 cm^{-1} . It is concluded that the helical conformation of the isotactic polystyrene mol. reflects mainly on the absorption bands associated with the modes of the groups composing the skeletal chain. H.H.H.

Colorants for PolystyreneE. A. Wick *Mod. Plastics*, 38 (10) (1961) 88-89, 180*Chem. Abs.*, 55 (21 Aug 1961) 17075

A table of recommended colorants. C.O.C.

Thermal Stability of Polydivinylbenzene and Copolymers of Styrene with Divinylbenzene and Trivinylbenzene

S. Straus and S. L. Madorsky

J. Res. Nat. Bur. Stand. A, 65 (May-June 1961) 243-248

Samples of polydivinylbenzene (PDVB) and copolymers of styrene with divinylbenzene (DVB) and trivinylbenzene (TVB) were pyrolysed in a vacuum at 346-450°C. With a copolymer containing 2% DVB, the yield of styrene monomer is somewhat greater than that obtained with pure polystyrene. Copolymers containing 25% DVB or TVB yield reduced amounts of styrene monomer and those containing about 50% DVB yield none. Rates of thermal decomposition of PDVB and the copolymers have been obtained in the range 330-390°C. The activation energy appears to increase as the amount of DVB or TVB in the copolymer is increased. W.R.M.

Pyrolysis of Fluorocarbon Polymers

L. A. Wall and S. Straus

J. Res. Nat. Bur. Stand. A, 65 (May-June 1961) 227-238

Studies of the thermal decomposition of fluorocarbon polymers suggest that completely fluorinated polymers are most stable. Studies of the decomposition of hexafluoropropylene telomers and of a copolymer of tetrafluoroethylene and hexafluoropropylene suggest, however, that the homopolymer of hexafluoropropylene should be unstable. The photo-induced decomposition of polytrifluoroethylene suggests that mutual termination

of radical intermediates occurs, and a diffusion effect on depropagation is pronounced below 250°C. W.R.M.

Gamma Irradiation of Fluorocarbon Polymers

R. E. Florin and L. A. Wall

J. Res. Nat. Bur. Stand. A, 65 (July-Aug 1961) 375-387

A number of fluorocarbon polymers were irradiated with γ -radiation at doses of up to 10^{22} ev/g. *G*-values were measured for volatile products, free radicals and, in the case of polychlorotrifluoroethylene, chain scission. Degradation or cross-linking was followed by measuring the tensile strength and zero-strength-time. The presence of air greatly accelerates chain scission of polytetrafluoroethylene and its hexafluoropropylene copolymer. The mechanism of radiation-induced changes is discussed in terms of free-radical intermediates. Perfluoroaromatic polymers may have superior radiation resistance. W.R.M.

Imparting Dyeing Properties to Polytetrafluoroethylene by Radiation Graft Copolymerisation

J. Dobo, A. Somogyi, and E. Lakner

Plaste u. Kautschuk, 8 (1960) 393-395*Chem. Abs.*, 55 (24 July 1961) 14969

Polytetrafluoroethylene film is given affinity for dyes by surface-radiation grafting with suitable polymers. There is little deterioration in mechanical properties. The most suitable polymers were vinyl acetate copolymers. Disperse dyes gave best results on the treated film. C.O.C.

Polyvinyl Glycollic Acid Ether (Carboxymethylation of Polyvinyl Alcohol)

M. Hida

J. Soc. Textile Cellulose Ind. Japan, 17 (July 1961) 669-675

Carboxymethylation of polyvinyl alcohol with sodium monochloroacetate in presence of caustic soda has been investigated, showing the dependence of the degree of etherification on the reaction conditions. In the early stages, in presence of excess of sodium monochloroacetate, the reaction has been identified as non-homogeneous pseudo-unimolecular. The rate of reaction is proportional to $[NaOH]$ and the activation energy is 14.8 kcal/mole. C.O.C.

IV—Mechanism of the Reaction*Ibid.*, 675-681

It is concluded that at first only the surface micelles of polyvinyl alcohol are carboxymethylated. Introduction of the $-OCH_2COONa$ grouping in this way causes swelling and with it an increase in $NaOH$ absorption with consequent increase in reactant penetration and reaction rate, the inner micelles then being affected. I.G.L.

PATENTS**Calendering Unit for Production of Uniform Grain- ing in Variegated Sheets**Armstrong Cork Co. *USP* 2,964,785 (30 June 1955)

Jaspé sheets or other masses of material to be consolidated are fed on a conveyor belt to the calender nip. About this belt there is a floating control member, preferably an endless belt, placed at an angle with respect to the plane of movement of the conveyor and free to tilt to and from the conveyor so as to engage the sheets in the conveyor and control their movement. This ensures proper pressure feeding of the sheets into the nip. C.O.C.

Crystalline Polyolefins having High Affinity for Dyes

Montecatini Società Generale per l'Industria Mineraria e Chimica

BP 875,758 (Italy 19 Feb 1957)

A high-mol.-wt. crystalline polypropylene, preferably after being peroxidised, is mixed with 1-25% of a monomer capable of undergoing free-radical polymerisation, e.g. styrene, and then heated to above the m.p. This causes the monomer to polymerise and be grafted on to the polypropylene. The product has good affinity for disperse and basic dyes, with which it yields dyeings of excellent fastness to washing and rubbing. C.O.C.

Cross-linking of Polyethylene containing Carbon Black (C.I. Pigment Black 6 and 7)

Anaconda Wire & Cable Co.

BP 875,734 (U.S.A. 26 June 1959)

Solid polyethylene pigmented with an alkaline Carbon Black has added to it a little of a quinhydrone dimeride,

e.g. a dimeride of *p*-benzoquinone and hydroquinone, and is then heated to a temperature above that at which the quinhydrone dimeride undergoes disproportionation into free radicals. The free radicals so generated cause cross-linking of the polyethylene chains. The products have excellent thermal stability, chemical, and electrical properties. C.O.C.

Dry, Free-flowing, Pigmented Polyvinyl Chloride Composition

Western Electric Co. USP 2,958,669 (30 Jan 1957)
Dry pigment is mixed with a plasticiser, heated if necessary, and the resultant paste slowly added to a powdered, dry blend resin so that the particles of resin absorb the plasticiser and become coated with the pigment. The product is used as a colour masterbatch. C.O.C.

Imparting Bacteria-repellency to Polyvinyl Chloride FBy

BP 875,355 (Germany 12 Mar 1959)
Incorporation of Zn pentachlorophenate and salicylanilide renders polyvinyl chloride lethal to bacteria and many other micro-organisms. Presence of the anilide increases the effect of the phenate and also suppresses the tendency of the latter to discolour the polyvinyl chloride. C.O.C.

Decorative Effects on Thermosetting Plastics

Johns-Manville Fiber Glass USP 2,959,511 (21 Jan 1955)
A liquid thermosetting resin is placed upon a thin synthetic-polymer film which is appreciably affected either by moisture or by the resin. A filler or reinforcement is then combined with the resin and a second film is placed on the combined resin and filler mix to form a wet lay-up. The assembly is then transferred to a mould and heat cured. During curing the films expand and crinkle uniformly, this uniform crinkled design being imposed upon the surface of the resin. C.O.C.

Foamed Styrene Polymers containing an Inorganic Pigment

Monsanto Chemical Co. USP 2,962,456 (6 Jan 1958)
Foamed styrene polymers are obtained by extending a free-flowing blend of a finely powdered styrene polymer and an inorganic pigment having adsorbed on the pigment surface < 25% of its weight of an aliphatic hydrocarbon of b.p. 10–80°C. C.O.C.

Polymethylene Polyphenols (III p. 563)

Coloured Synthetic Resin and Dyes therefor—Moulding Powders and Pigments (IV p. 571)

Colourless Carbon Papers and Heterocyclic Compounds therefor (IV p. 574)

Carbon Black (C.I. Pigment Black 6 and 7) Slurries (IV p. 575)

Polymer Deposition in Polyvinyl Alcohol Fibres (VI p. 578)
Dye-receptive Graft Copolymers (VI p. 579)

Polysulphonamides (VI p. 579)

Peroxide Bleaching of Linoleum (VII p. 580)

Mass-coloured Polypropylene (VIII p. 583)

Elastic Coating and Impregnating Compositions Compatible with Daylight Fluorescent Pigments (X p. 587)

Grafted Synthetic Addition Polymers (X p. 588)

XIV—ANALYSIS; TESTING; APPARATUS

Holmium Filter for Checking the Wavelength Scale of Recording Spectrophotometers

J. M. Vandenberg

J. Opt. Soc. Amer., 51 (July 1961) 802–803
A number of spectral line sources have been used to calibrate the wavelength scales of spectrophotometers in the visible and ultraviolet, e.g. the mercury vapour arc and several types of discharge tube, as well as secondary standards, e.g. the didymium filter. Holmium oxide is suggested as a suitable calibration standard. The absorption spectrum of a 1% solution of the oxide in perchloric acid (5-cm cells) is shown to give 15–20 sharp bands in the visible and ultraviolet. Melts of holmium oxide in ultraviolet transmitting glass, Corning CS3–138 (glass code

3130) are described, calibrated specimens of which are obtainable from the Photometry and Colorimetry Section of the National Bureau of Standards. E.COATES

Use of Dead-stop Method in Automatic Titrations and Concentration Control in the Textile Industry

I. Ruzsánák and G. Lepénye

Magyar Kém. Lapja, 16 (1961) 143
Chem. Abs., 55 (21 Aug 1961) 17014
NaOCl was titrated with stabilised Na₂SO₃ dissolved in 0.1-N acetic acid containing 5 g/l. KI, and Na₂SO₃ was titrated with NH₄Cl in presence of KI. In both cases the end point was indicated by current change caused by the first drop of I₂. Both series of results agreed and showed the method to be accurate even in 0.001-N concentrations. C.O.C.

An Optical Method for the Determination of Diffusion Coefficients

A. van Tets and Hs. H. Günthard

Helv. chim. Acta, 44 (1 Aug 1961) 1356–1364
A method for the determination of surface and bulk diffusion coefficients based on data for optical density on thin slabs is discussed, and its limitations are considered. The mathematics involved includes the use of time derivatives of optical density, numerical Laplace transformation, and successive approx. by least squares. Explicit formulae and numerical tables and curves for the relevant functions which might be useful in practical applications of the method are included. H.H.H.

Spectrophotometric Determination of Naphthylamines

H. O. Spauschus *Anal. Chem.*, 33 (Sept 1961) 1334–1337

The sample is dissolved in a halogenated solvent and exposed to ultraviolet irradiation. A colour develops rapidly and obeys the Beer–Lambert law for the range 10–100 p.p.m. By this means naphthylamines and their halogen-substituted derivatives are determined. P.B.S.

Detection of Traces of Surface-active Agents

L. Meckel and U. Krause

Textil Praxis, 16 (June 1961) 595–596
A modification of the Weber reagent (tylose + sulphuric acid) in which the tylose is replaced by vanillin can be used to detect small amounts of surface-active agents. The latter are made up as approx. 1% aqueous solutions, e.g. by extracting from a fabric with alcohol, evaporating, and dissolving in water. This solution (0.5 ml) is shaken with an equal volume of a satd. aqueous solution of vanillin, and conc. sulphuric acid (2 ml) is carefully added without mixing to form a separate layer. The colour formed at the interface is observed immediately, after 3 min, and after 10 min. The rate of colour development, the hue, and the appearance under ultraviolet radiation give information about the type of surface-active agent present. Polyglycol ethers produce a cherry-red ring which turns bluish, but to obtain some indication of the type of compound involved a direct comparison with known materials must be carried out. Propanol and higher alcohols produce a colour, but methanol, ethanol, and glycerol do not. The limit of sensitivity in mg per ml for characteristic colour identification, and for detection only, is, respectively: alkyl sulphate (47%) 0.1, 0.01; aralkyl sulphonate (82%) 1.0, 0.1; alkyl sulphonate 0.5, 0.01; fatty-acid derivative (80%) 0.1, 0.01; polyglycol ether 0.25, 0.025; soap 0.25, 0.025. Soap interferes but can in most cases be removed by precipitation with 2% calcium chloride and filtration before the filtrate is tested. E.C.

Determining Iron in Syntans

Z. Hrabal

Kořařství, 10 (1960) 314–316
Chem. Abs., 55 (21 Aug 1961) 17057
Fe was determined colorimetrically in syntan ash by KCNS or by a complexometric method using Tiron (di-Na 4,5-dihydroxy-*m*-benzenedisulphonate, Atkinson and McBryde, *Chem. Abs.*, 52 (1958) 959). Both methods are exact, but the Tiron method is much simpler and quicker. C.O.C.

Determination of Fluorescent Brighteners

L. Meckel

Textil Praxis, 16 (July 1961) 737–738
A method is described for the characterisation of fluorescent brighteners by means of paper chromatography. H.H.H.

Chromatography of Dye Intermediates. XII—Chromatographic Separation and Identification of 2-Naphtholsulphonic Acids

J. Latinák

Collection Czechoslov. Chem. Commun.,

26 (1961) 403–415 (in German)

Chem. Abs., 55 (7 Aug 1961) 15935

Chromatographic properties of all the isomeric acids were studied in 4:1:5 butanol-acetic acid-water and 2:1 propanol-aq. NaHCO_3 on normal paper and on paper impregnated with 5% NaHCO_3 . Higher-sulphonated derivatives were converted to monosulphonic acids by splitting off SO_3H groups and were identified as such. Complicated mixtures were separated in the form of iodine derivatives. 2-Naphthol-3,8-disulphonic acid and 2-naphthol-3,5-disulphonic acid were found in the mixture obtained by sulphonating 2-naphthol. Relations between paper-chromatographic behaviour and chemical structure are discussed.

C.O.C.

Polarography of Basic Dyes

J. Thurel and B. Drevon

Ann. fals. et expert. chim., 54 (1961) 12–16

Chem. Abs., 55 (21 Aug 1961) 17009

Describes a method for characterising and determining di- and tri-phenylmethane dyes. The polarographic cell was contained in a methanolic soln. of the dye (100 γ /0.1 ml) and a KHPhtphthalate buffer, pH 5, prepared according to Clark and Lubs, the temperature being kept at $25 \pm 0.2^\circ\text{C}$. Dissolved oxygen was eliminated by bubbling N_2 , previously washed by alkaline pyrogallol, through the solution. Malachite Green (C.I. Basic Green 4), "hexamethyl violet", and Auramine O (C.I. Basic Yellow 2) could be detected in an equimolecular mixture of the dyes. Malachite Green and hexamethyl violet (20–100 γ in 5 ml soln.) were determined, the height of the polarographic curves obtained being proportional to the concentration of dye.

C.O.C.

Reductometric Estimation of Hydroxytriphenylmethane Dyes with Vanadium (II) Sulphate

M. Matrká and Z. Ságner

Chem. průmysl, 11 (1961) 135

Chem. Abs., 55 (7 Aug 1961) 15935

A mixture of Aurine (C.I. 43800) (0.3 g), alcohol (50 ml), water (100 ml), and conc. H_2SO_4 (5 ml) was heated to 50°C for 1 min in a closed vessel provided with a burette, thermometer, and inlet and outlet for N_2 . After addition of 25 mol 0.1-N VSO_4 , the mixture was cooled to 20°C and titrated with 0.1-N $\text{Fe}(\text{NH}_4)(\text{SO}_4)_2$ up to weak violet colouring of Safranin T (C.I. Basic Red 2) (0.2% soln). Throughout the operations N_2 passed through 0.1-N VSO_4 was bubbled through the mixture. The 0.1-N VSO_4 was stored under hydrogen. Accuracy was $\pm 0.50\%$.

C.O.C.

Additional Acid-Base Indicators in Glacial Acetic Acid

O. W. Kolling and T. L. Stevens

Anal. Chem., 33 (Sept 1961) 1384–1387

Dyes of the azine, acridine, thiazine, and thiazole classes are evaluated for their suitability as acid-base indicators in glacial acetic acid medium. Six dyes show distinct and reproducible colour changes, detectable visually.

P.B.S.

Paper Chromatography of Bixin (C.I. 75120) and Related Compounds

G. G. McKeown

J. Assocn. Offic. Agric. Chem., 44 (1961) 347–351

Chem. Abs., 55 (21 Aug 1961) 16839

A method is given for separating bixin and related cpd. Labile (cis-form) bixin, labile norbixin, labile methyl bixin, and their corresponding (trans-form) isomers were studied. R_f and R values relative to those of Oil Yellow AB (C.I. 11380) are given. Extracts of *Bixa orellana* seeds (C.I. Natural Orange 4) and commercial Annatto (also C.I. Natural Orange 4) butter and cheese colours were examined for their constituent colorants. Stable labile bixin (cis-form) was the chief colorant in precipitated material in Annatto butter colours, and labile norbixin in cheese colours.

C.O.C.

Extraction of Acid Dyes from Confectionery by a Quaternary Ammonium Compound

J. Vollaire-Salva

Ann. fals. et expert. chim., 54 (1961) 17–20

Chem. Abs., 55 (10 July 1961) 13706

Gums and pectins are extracted with acetone or alcohol.

The sample is then brought to pH 9 with aq. Na_2CO_3 and hexadecylcyclohexyldimethylammonium bromide and CHCl_3 added. The CHCl_3 solution is extracted 3–5 times with conc. HCl , the dye dissolved in dil. NH_4OH and this solution chromatographed.

C.O.C.

Extraction and Determination of Carotene (C.I. 75130)

J. Dick and S. Rusu-Bodreanu

Acad. ref. populare Romine, Baza cercetării științ.

Timișoara, Studii cercetării științ.

Ser. științe chim., 7 (1960) 61–67

Several rapid methods of extraction and determination are reviewed, and a shortened method is suggested.

C.O.C.

Identification of Fibre Blends by Infrared Spectroscopy

M. K. Wharton and F. H. Forziati

Amer. Dyestuff Rep., 50 (10 July 1961) 515–518

A method for the qualitative analysis of fibre blends by infrared spectroscopy is described. The coarsely powdered blend is allowed to separate into components in a density gradient column. Each component is removed with a hypodermic syringe and, after drying, an infrared absorption spectrum is obtained using the KBr pellet technique. The spectra provide rapid and positive identification of the fibres as to chemical or generic class, but supplementary techniques are sometimes needed to differentiate between fibres of the same class.

S.B.D.

Identification of Synthetic Fibres in Blended Yarns

G. Vyskocilová-Janáková and K. Schwertasek

Textil Praxis, 16 (Aug 1961) 809–812

This is a group-analytical scheme based on solubility in a range of solvents. Proof of dissolution is by reprecipitation.

J.W.D.

Sample Preparation of Fibres for Identification by Infrared Spectroscopy

S. Burgess and H. Spedding

Chem. and Ind., No. 30 (29 July 1961) 1166–1167

Samples of cellulose fibres, nylon 6.6, Terylene, Orlon, and Courtelle may be prepared for infrared (I.R.) spectroscopy in the form of fibre-films. A bundle of fibres cut into 1–2-mm lengths is rubbed, in the form of a ball, through wire mesh (200–400 mesh/in²) directly on to the pressing surface of a die and subjected to a pressure of 55 tons/in² for 5 min. The resultant fibre-film may then be used for qualitative I.R. analysis and identification. The method overcomes the disadvantages of undesirable absorption by the oil and the presence of adsorbed water in the mull and pellet methods, respectively. By this means the spectra of secondary cellulose acetate and triacetate can be differentiated in the 3μ residual hydroxyl band without interference from the overlapping adsorbed water band.

F.J.

Staining of Cellulose Fibres and Foil with Triphenyltetrazolium Chloride

O. Szaboľcs Melland Textilber., 42 (July 1961) 796–798

A new chemically reactive staining method for cellulose is described and compared with some conventional methods. The triphenyltetrazolium chloride forms a dye by reaction with the reducing carbonyl groups of the cellulose. The method permits the core and the cuticle of a fibre to be clearly distinguished, even in the case of "full core" (stained red) and "full cuticle" (stained yellow) fibres. The method is simpler to use than existing methods.

P.B.S.

Determination of the Cuprammonium Fluidity of Lignified Fibres

F. S. Young J. Textile Inst., 52 (July 1961) T324–T329

This method for fibres such as sisal and jute is based on the corresponding test for cotton (B.S. 2610:1955) modified to allow for the large proportion of the material insoluble in cuprammonium hydroxide. Pyrogallol is added to enable the solution to be filtered in the presence of air before the fluidity is determined, and the dry residue is weighed to give the amount of soluble material by difference. A standard curve of fluidity against concentration is prepared. The fluidity is an index of chemical degradation. The amount of insoluble material decreases with increasing degradation.

J.C.F.

Quantitative Determination of Wool in Wool-Acrylic Fibre Mixtures

G. Ammacher

Melliand Textilber., 42 (July 1961) 822-824

Available methods are reviewed and two further methods described. Boiling the material for 15 min with 2.5% potassium hydroxide, which dissolves the wool, is a simple indirect method. All the other methods depend upon the dissolution of the acrylic fibre in a solvent (dimethylformamide, Sesolvan NK, or formic acid-zinc chloride solution) and direct weighing of the wool. Four of the methods are compared experimentally. P.B.S.

Determination of Fatty Matter in Scoured Wool and Tops

R. P. Harker

II—Investigation of the Refractive Index Method for the Determination of Combing Oil in Solvent Extracts from Tops

J. Textile Inst., 52 (July 1961) T309-T316

The refractive index method has been critically examined, and the present modification makes allowance for interference from constituents other than combing oil. The method is suitable for process control: appropriate standards must be available, and samples of unknown origin cannot be analysed with confidence. Accurate temperature control during the measurement of refractive index is essential.

III—Evidence for the Existence of a Barrier to Non-polar Solvents

Ibid., T317-T320

Extraction experiments with different solvents on scoured wool and tops have indicated the existence of a barrier to non-polar solvents. A mechanism for the formation of such a complex layer is proposed by analogy with the phase separations observed with soap-detergent-water systems. Direct evidence for such a barrier has been obtained by extraction experiments on macroscopic grease layers. J.C.F.

Bromine Absorption Method for Determining the Iodine Value of Fatty Extracts of Wool

R. S. Hartley and B. C. Hobson

J. Textile Inst., 52 (July 1961) T321-T323

This method, which is based on the increase in weight when the sample is treated with a solution of bromine in carbon tetrachloride, requires only 0.02-0.05 g of fatty matter. It can be used in conjunction with the W.I.R.A. rapid-extraction apparatus. J.C.F.

Determination of Linear Density of Textile Fibres by Weighing

British Standards Institution

BS 2016:1961

A revision of the standard originally published in 1953. Linear density is defined as "mass per unit length of a fibre" or filament expressed in *millitex* (mg/km). Four methods of determining linear density are described, two of which are intended for fibres cut to a definite length (including continuous filament) and two for whole fibres. More rapid estimations of fibre fineness may be made by the methods of BS 3181 and 3183 but for research purposes the present method may be preferable. For wool fibres, fibre fineness is more usually measured in terms of fibre diameter, the method of BS 2043 being appropriate. M.T.

Determination of Tensile Properties of Individual Textile Fibres and Filaments

British Standard Institution

BS 3411:1961

A method based on T.T.S. No. 61 (*J. Textile Inst.*, 50 (Dec 1959) P776-P782). M.T.

Measurement of Equivalent Pore Size of Fabrics (Bubble Pressure Test)

British Standards Institution

BS 3321:1960

A method based on T.T.S. No. 59 (*J. Textile Inst.*, 50 (Dec 1959) P763-767). M.T.

Determination of the Stiffness of Cloth

British Standards Institution

BS 3356:1961

A method based on T.T.S. No. 60 (*J. Textile Inst.*, 50 (Dec 1959) P772-P775). M.T.

Rapid Photographic Method for Evaluating Bleaching Agents

R. L. Liss and T. B. Hilton

Chem. Specialties Mfrs. Assoc., Proc. 47th Ann. Meeting, (Dec 1960) 118-122*Chem. Abs.*, 55 (21 Aug 1961) 17014

A rapid photographic method is described which eliminates elaborate soiled-cloth preparations, and stains can be prepared for testing by preparing aqueous solutions or dispersions. Satisfactory correlation was obtained using artificially stained fabrics. C.O.C.

Gas-Liquid Chromatography of Linear Detergent Alkylates

J. M. Blakeway and D. B. Thomas

J. Chromatography, 6 (July 1961) 74-79

Two commercial samples of linear aryl alkylates used in the preparation of alkylaryl sulphonate detergents have been subjected to gas-liquid chromatography. Apiezon L grease was found to be a suitable selective liquid substrate. Substantially complete identification of the components of the commercial samples has been achieved. Retention vol. relative to n-hexadecane are given for all cpd. found, and the predominating fractions in both samples were those containing C₁₀-C₁₃ alkyl chains, the aryl group being substituted in positions 5 and 6 along the chain. Primary alkylbenzenes were absent. F.J.

Proximity Factor in the Judd Colour Difference Formula

A. C. Traub and I. Balinkin

J. Opt. Soc. Amer., 51 (July 1961) 755-760

The problem of representing colour difference in physical space is presented. The difference between lightness and luminous reflectance is discussed in connection with the establishment of an equal-lightness interval scale. The two attributes of visual sensation, hue and saturation, are considered together as chromaticness, each value of which is represented by a pair of chromaticity co-ordinates. The division into equal chromaticness intervals is achieved by observer estimate. Exact uniformity of the chromaticness surface cannot be achieved on a surface of zero curvature, but a chromaticness plane is accepted as being approximately uniform. The difficulties in correlating lightness difference (ΔL) intervals with chromaticness difference (ΔC) intervals are discussed, and the effect of the proximity of observed pairs is indicated. The greater effect of separation is on lightness, and a given colour space can have meaning for samples of fixed size at one separation only. As the separation is changed so must the entire colour solid shrink or stretch along the lightness axis. The Judd colour space is described and it is on this basis that experimental results are represented,

$$(\Delta E)^2 = (\Delta C)^2 + (\Delta L)^2 \\ = (k_2 \Delta^2 S)^2 + (k_1 \Delta(A^{\frac{1}{2}}))^2$$

where ΔE is the nett colour difference, A is the luminous reflectance and ΔS a rectilinear length in the Judd U.C.S. triangle. For samples in close contact, $k_1 = 120$, reducing to 30 for wide separation. A value of $k_2 = 600$ is taken so as to give $\Delta E = 1$ for colour differences large enough to be perceptible but small enough to be negligible commercially. The experimental details are given for the measurement of perceptible differences in sample pairs, which were ultimately obtained by controlled exposure of photographic paper. The samples were 1-cm squares, side by side on a dark ground of 5% luminous reflectance, viewed with unrestricted binocular vision at a distance corresponding to an angular field of 2°. The illuminance was approximately 20 ft-c. It is shown that

$$k_1(\theta) = 30 + [18/(0.2 + \theta^{\frac{1}{2}})]$$

for samples with an angular separation of θ degrees.

E.COATES

Light Absorption of Dyes. IV—Relation of Transmission and Reflection of Light by Diffusing Materials to Concentration of Colour

C. H. Giles, S. M. K. Rahman, and D. Smith

Text. Research J., 31 (Aug 1961) 679-687

Beer's law is not usually obeyed by the dye present in dyed materials, probably because of the adsorbed dye aggregates present. It is, in fact, only obeyed by a true solution or suspension of particles of constant size

distribution. Application of Beer's law to transmission and reflection systems is discussed, and a more general relation obtained by substituting for c (colour concentration factor in Beer's law equation) the empirical factor $c/(1+ac^2)$ where a and x are constants (probably functions of the difference in refractive index between the dye and surrounding medium) and x is a simple fraction. With a dyed viscose rayon fabric, about 20% of the reflected light was specularly reflected from the surfaces of lightly dyed fibres. A linear relationship between the logarithm of the colour concentration of a diffusely reflecting coloured surface and its reflectance density was derived as a simple extension of Beer's law and was confirmed experimentally on a variety of dyed fabrics. C.O.C.

Objective Evaluation of Colour Differences

— Kling and J. Kurz

Textil Praxis, 16 (July 1961) 733-737

A suitable colour scheme and accurate difference formula are described which enable quant. measurements of colour differences to be made. There are 9 diagrams. H.H.H.

Colour-matching Lamps—an Enquiry into Members' Wishes

K. McLaren

J.S.D.C., 77 (Sept 1961) 435-437

Determination of Fastness to Daylight of Coloured Textiles

British Standards Institution

BS 1006:1961

This method is identical with that described in the Society's publication *Standard Methods for the Determination of the Colour Fastness of Textiles* (Second Edition) M.T.

Photometric Determination of Dye Fastness

D. D. M. Casani and L. D'Arja

Boll. sci. fac. chim. ind. Bologna, 19 (1961) 11-20
Chem. Abs., 55 (21 Aug 1961) 17014

The fastness of dyes on cloth can, with the exception of that to light, be expressed as the ratio between luminosity increment caused by degradation during the test and the difference between the luminosities of the undyed fabric and the dyed fabric before testing. Values for the calculations were obtained by measuring the areas underlying the specific reflectance curves in the 400-700 $m\mu$ region obtained with a spectrophotometer or by colorimetric measurement. A relation was established between the indexes obtained by the photometric methods and those derived visually by direct comparison with the grey scale. For all the dyes examined (on nylon, cotton, and wool), with the exception of yellow dyes, photometric results agreed with visual measurements. C.O.C.

Objective Evaluation of Light-fastness Test Results

P. J. Holzer *Melliand Textilber.*, 42 (July 1961) 811-815

A description of the Fade-Ometer and Weather-Ometer light-fastness machines and their operation is given. Using these machines, the German and Soviet light-fastness standards were compared over various exposure times. Results are given in tables and as graphs, of fading, colour value, wavelength, and brightness, at 24-h intervals. An approximate relation between the German and Soviet fading scales is also given. P.B.S.

Critical Comparison of the Data afforded by Two Different Rubbing-fastness Test Meters

O. Blum

Textil Praxis, 16 (July 1961) 725-728

Details are given of the Crockmeter and of the Ruf instrument for measuring fastness to rubbing, and data are recorded for 56 samples of various fibres. Comparison shows that agreement between the two instruments was excellent in most cases. H.H.H.

Resolution of Textile Faults by Microscopic Examination

A. Kapp and K. Mahall

Textil Praxis, 16 (June 1961) 600-607

Samples of resin-treated nylon fabric showed a wrinkling not evident in a similar Perlon fabric. Microscopic examination of prepared sections showed that in the faulty fabric the resin had coated the threads without penetration. Variation in stiffness across another sample of Perlon fabric was shown by microscopic examination to be due to co-fusion of the threads in the stiff parts and

was hence attributable to temperature variation across the setting oven. A cotton poplin damaged with small holes on treatment with 15% caustic soda showed split and ragged fibres on the margins typical of acid damage. Various samples of dyed cotton fabrics showing streakiness were found to contain mechanically damaged threads, and banding effects were found to be due to the use of a mixed cotton-viscose rayon yarn in certain parts of the fabric. Streakiness in a blue cotton locknit fabric was shown to be due to unlevel mercerisation of the yarn, detected by variation in the convolution and cross-section of the fibres in different lengths of yarn.

Effectiveness of sizing processes on two yarns was compared by preparing sections, staining the starch size with iodine, and microscopic examination. Loss of strength in a hemp yarn showing dark specks was attributed to mould infection.

Streakiness in the warp of a yarn-dyed fabric with brown warp and blue weft was found to be due to variations in the warp yarn. Streakiness and small light specks in a lining material with acetate warp and viscose weft was found to be due to local displacements of warp and weft in the lighter areas, attributable to slipperiness of the warp. Similar defects in a corset fabric with a rayon-covered rubber warp and Perlon weft were attributable to non-uniform disposition of the weft on the warp. Light specks in a military union cloth after storing in the roll for a week were traced to bacterial or fungal damage of the wool. Small dark friable deposits on a woollen yarn which was hank-dyed in plant used for both wool and synthetic-polymer fibres were shown to be degraded wool. Examination of the dyeing plant showed that wool fibres lodged in certain corners of the plant and were degraded by the alkali-treatment of synthetic-polymer fibres and could be deposited on the yarn during dyeing. Many combed-yarn fabrics show weft irregularities which become apparent under damp conditions and this is usually due to the use of a weft of variable moisture content in weaving setting up uneven tensions in the cloth. The text is supported by photomicrographs of many of the faults discussed. E.C.

Crease-recovery Test for Fabrics

S. L. Anderson and G. E. Settle

J. Textile Inst., 52 (June 1961) T298-T299

A method for the determination of the recovery of fabrics from creasing which can be applied to very limp fabrics, e.g. wool mixture fabrics, has been developed. It is based on BS 3086:1959, but a shorter specimen length is taken and a 10 \times projector used to show up the profile of the test specimen. P.G.M.

Behaviour of Antistatic-finished Textiles in Wearing and Washing Tests—III

E. Wagner

Chemiefasern, 11 (July 1961) 477-482

Washing and wearing tests have been carried out on polyester, polyamide, and polyacrylonitrile fabrics. Details of methods employed are given. Maximum field strength obtainable by rubbing and the course of the fall in field strength with time have been determined, using one permanent antistatic agent and two easily removable by washing. The course of the fall in field strength is dependent on the chemical constitution of the antistatic agent. The presence of hydrophilic groups and orientation of the molecules of the agent on the fibre surface increase the fall in field strength and with it the electrical conductivity. Relative humidity (30-60%) has been found to influence considerably the extent to which an antistatic-treated fabric can be charged, but has scarcely any effect on the rate of loss of charge. With untreated fabrics the reverse is the case. Ease of removal of the antistatic agent is dependent on its chemical constitution. With the two easily removable agents, practically all antistatic action is lost after only one washing treatment. The carrier is of some importance with regard to the washing fastness of the permanent agent. Washing experiments on deliberately soiled textiles showed that the antistatic agent can, to a very slight extent, hinder or assist the washing action. Take-up of dirt can be assisted or hindered by the presence of the antistatic agent—dirt repellency or attraction is not a specific property of any particular antistatic product, nor is the ability to influence the efficiency of dirt removal by washing. It is concluded that the behaviour of antistatic-treated fabrics can be

comprehended only under actual conditions of use and that it is impossible to assign universal properties to any particular antistatic agent. I.G.L.

Occurrence of Pyrrole-2-carboxylic Acid in Hide Hydrolysates and Its Bearing on the Determination of Hydroxyproline

C. Deasy

J. Amer. Leather Chem. Assoc., **56** (July 1961) 357-361
A ninhydrin-positive substance found in chromatograms of hydrolysates of certain dinitrophenyl collagen fractions has been identified as pyrrole-2-carboxylic acid (I); it is probably formed by decomposition of a sialic acid of the hide during the acid hydrolysis. Since I reacts with *p*-dimethylaminobenzaldehyde to give a coloured product that absorbs at 560 m μ , it will contribute to the apparent amount of "hydroxyproline" found by the Neuman-Logan procedure. The small amount present in hide hydrolysates has been found to be without significant effect, but in samples with high sialic acid and relatively low hydroxyproline contents it might become significant. J.W.D.

Design of Methods for Testing Fastness of Light Leathers to Rubbing Under Various Conditions

D. Grimwade and S. Wolstenholme

J. Soc. Leather Trades Chem., **45** (July 1961) 276-290
Development of the above Society's Official Methods for measuring the fastness of light leathers to dry rubbing, to wet rubbing, and to rubbing when wetted from the back with organic solvents is described. Attention is drawn to the work of the International Fastness Tests Commission with the Crockmeter, and a comparison of the Crockmeter with the S.A.T.R.A. machine accepted by the Society is given in detail. J.W.D.

Design of a Method for Testing Fastness of Coloured Leathers to Marking Off

C. F. Kirkpatrick

J. Soc. Leather Trades Chem., **45** (Aug 1961) 318-321
Adaptation of an earlier form of the test, considered by the Committee on the Fastness of Dyes on Leather in 1949 (*ibid.*, **33** (1949) 238), to yield the present Official Method of the Society. Experimental results are quoted. J.W.D.

Examination of Rub Test for Determination of Fastness of Leather Finishes

A. W. Landmann, R. G. Mitton, and F. R. Morgan

J. Soc. Leather Trades Chem., **45** (July 1961) 291-299
An account of work carried out by the British Leather Manufacturers' Research Association which led to suggestions for the improvement of the reproducibility of the rub-fastness test; these were included in the above Society's Official Method. J.W.D.

Selection of Solvents for Paper Chromatography of Lacquer Resins

K. Weigel

Deutsche Farben-Zeitschrift, **15** (July 1961) 284-287
In this concluding article on initial sorting tests on solvents for the separation of lacquer resins, results are given of the behaviour of various solvents in eluting resin spots on strips of paper suspended inside uncovered 100-ml glass cylinders using 10 ml of solvent. Results tend to be diffuse owing to the range of molecular weight present in most resins. Benzene and xylene are effective in separating butylated melamine resins from urea resins, but esters, ketones, glycol ethers, chlorohydrocarbons, and aqueous acetone are incapable of differentiating these resins. Ketone resins elute well with ketones but not with esters, alcohols, or glycol ethers. They can be separated from melamine lacquer resins by elution with benzene hydrocarbons, but benzene, xylene, and chlorohydrocarbons effect no separation. E.C.

PATENTS

Temperature-measuring Device Especially Suited for Measuring the Surface Temperature of Rotating Cylinders

British Cotton Industry Research Assocn.

BP 875,025 (1 Apr 1959)

An instrument especially suitable for use where the temperature range is 50-150°C includes a flexible thermocouple which conforms to the curvature of the cylinder and having, at the side touching the cylinder, a thin layer of low-friction material, e.g. polytetrafluoroethylene. C.O.C.

Measurement of Oxidation-Reduction Potential

Diamond Alkali Co.

BP 875,140 (U.S.A. 7 Apr 1959)

Describes an oxidation-reduction potential cell for use in halogenation, particularly chlorination, reactions, e.g. in bleaching paper pulp. C.O.C.

Auxiliaries for Prevention of Gas-fume Fading of Dyed Cellulose Acetate Fibres (III p. 563)

Rapid Colorimetric Determination of Crystal Violet Base (IV p. 566)

Permanent Pleating of Polyester/Wool Mixtures (X p. 586)

XV— MISCELLANEOUS

A Simple Method for Determining Rate Constants and Orders of Reaction

R. W. Wilkinson

Chem. and Ind., No. 35 (2 Sept 1961) 1395-1397

The following equation, derived from the general expression connecting the extent, time (*t*) and order of reaction (*n*) other than first order, $t/p = 1/K + nt/2$, may be used to determine the rate constant (*K*) and order of any reaction up to 3; *p* is the fraction completed. Plots of *t/p* against *t* are straight lines for all orders, the slope yielding *n* and the intercept *K*. The rate of decomposition of benzene diazonium chloride at 30°C is given as one example using this method, the slope, found to be 0.54, giving *n* = 1 and the intercept, 2370 (\pm 30) s, giving $K = (4.22 \pm 0.05) \times 10^{-4} \text{ s}^{-1}$. A second-order reaction is exemplified by the pyrolysis of acetaldehyde at 518°C. F.J.

The Technical Heating Requirements of the Textile Industry

W. Sennhauser

Textil-Rund., **16** (July 1961) 364-372

A survey is made of methods of heat production and its economics in the textile industry, i.e. heating installations, heat transfer, heat storage, waste heat recovery, etc. P.B.S.

The Newer Fibres in the United Kingdom and Western Europe

A. Caross

J. Textile Inst., **52** (Aug 1961) P331-P342

The author, who is Overseas Director of I.C.I., reviews the prospects for synthetic fibres in Western Europe, and forecasts that consumption will treble by 1970, equalling the U.S. consumption. He believes that Western Europe will one day become a single free-trade area and that the British fibre makers will be able to compete in Western Europe at least on equal terms. W.G.C.

PATENT

Blackening of Spider Web Filaments, Adsorptive and Microscopically Etched Materials

Northrop Corpn.

USP 2,958,612 (10 June 1957)

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ERRATUM

Printing and Dyeing Pile Fabric. The abstract with this heading (*J.S.D.C.*, **77** (Sept 1961) 467) refers to the patents BP 868,415 and 868,416, and not to BP 865,415 and 865,416 as printed.

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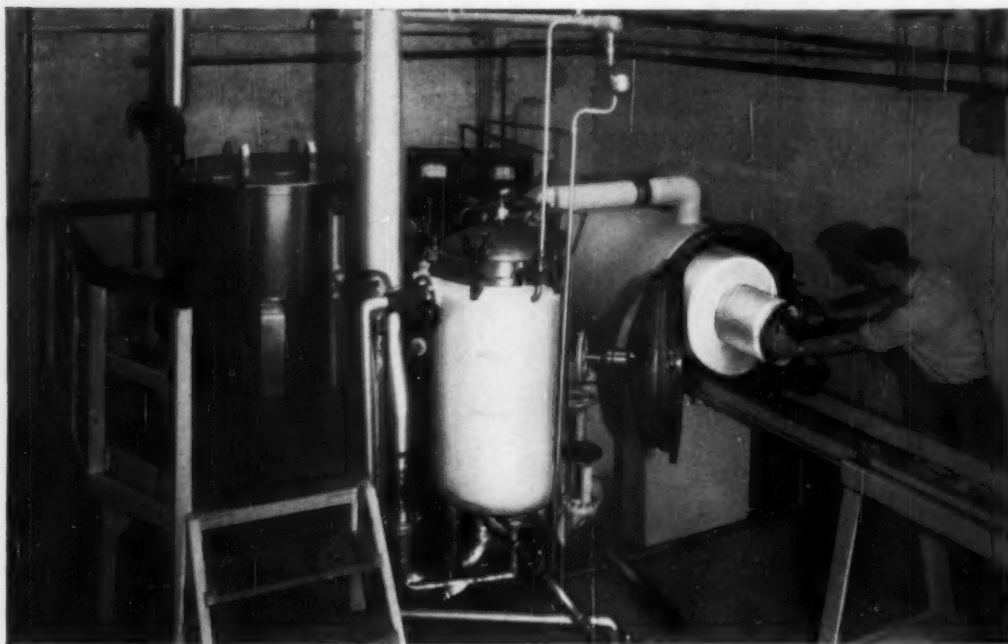
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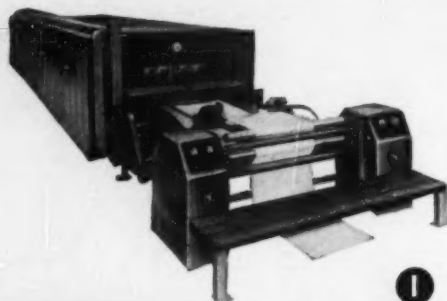
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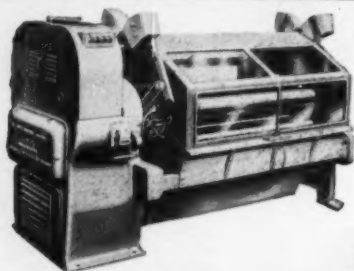
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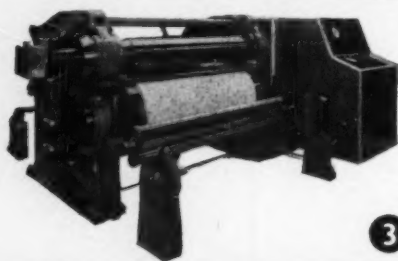
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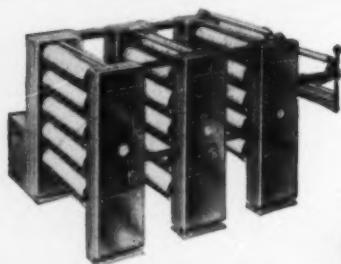
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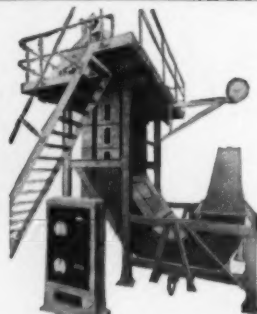
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